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A PRACTICAL TREATISE

ON

# ANIMAL AND VEGETABLE FATS AND OILS:

2

## COMPRISING BOTH FIXED AND VOLATILE OILS,

THEIR PHYSICAL AND CHEMICAL PROPERTIES AND USES, THE MANNER
OF EXTRACTING AND REFINING THEM, AND PRACTICAL
RULES FOR TESTING THEM;

AS WELL AS THE

MANUFACTURE OF ARTIFICIAL BUTTER AND LUBRICANTS, ETC.,

WITH LISTS OF AMERICAN PATENTS RELATING TO THE EXTRACTION, RENDERING,
REFINING, DECOMPOSING, AND BLEACHING OF FATS AND OILS.

BY

**WILLIAM T. BRANNT,**

EDITOR OF THE "TECHNO-CHEMICAL RECEIPT BOOK," "PETROLEUM," ETC.

SECOND EDITION, REVISED, AND IN GREAT PART RE-WRITTEN.

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# ANIMAL AND VEGETABLE FATS AND OILS.

## PART I.—CONTINUED.

## FIXED FATS AND OILS.

## CHAPTER XV.—CONTINUED.

### DESCRIPTIONS, PROPERTIES, ADULTERATIONS, ETC., OF OILS AND FATS.

### 2. *Glycerides of Linoleic Acid, or Drying Oils.*

### OILS OF THE VEGETABLE KINGDOM ONLY.

### *Zygophylleæ—Olacineæ (Terebinthinæ).*

1. *Coula-nut oil*, from the fruit of *Coula edulis*, H. Baillon, a plant of the west coast of Africa. The seed contains 40 to 50 per cent. of an excellent table oil; it, however, does not possess perfect drying properties.

2. *Xymenia gabonensis*, Roxb., grows also on the west coast of Africa. The seed contains 60 to 70 per cent. of oil, which is used in the manufacture of soap.

3. *Zachun oil*, from the seed of *Balanites Roxburghii*, Planch., a tree indigenous to Africa. It is used by the natives for many purposes.

### *Euphorbiaceæ (Tricoccæ).*

1. ***Candle-nut oil***, or ***bankoul-nut oil*** (*oleum aleurites; Bankulnussöl*, G.; ***huile de noix de bankoul***, F.), from the seed of ***Aleurites moluccana***, Willd., a tree growing everywhere in Oceanica

and the Sandwich Islands, and also cultivated in India, South America, the West Indies, etc. Though the oil has been used for many years by the natives, its general use is of very recent date.

The seed contains from 58 to 65 per cent. of oil.

| | *a.* Indian seed. | | *b.* Tahitian seed. | |
|---|---|---|---|---|
| Oil. | 59.82 p. c. | | 62.15 p. c. | |
| Organic substance | 31.53 " | | 29.50 " | |
| protein substances therein. | | 23 p. c. | | 22.50 p. c. |
| Ash | 3.50 " | | 3.35 " | |
| Water | 5.15 " | | 5.00 " | |
| | 100.00 " | | 100.00 " | |

The oil cake contains:—

| | *a.* Indian seed. | | *b.* Tahitian seed. | |
|---|---|---|---|---|
| Oil. | 8.93 p. c. | | 9.20 p. c. | |
| Organic substance, | 75.04 " | | 74.24 " | |
| protein substances therein. | | 52.00 p. c. | | 51.70 p. c. |
| Ash | 8.96 " | | 9.36 " | |
| Water | 7.07 " | | 7.20 " | |
| | 100.00 " | | 100.00 " | |

The ash contains the following constituents:

| | Candle nuts. | Candle nut cake. |
|---|---|---|
| Potash | 17.25 per cent. | 20.30 per cent. |
| Soda | 0.42 " | 1.30 " |
| Magnesia | 15.13 " | 16.26 " |
| Lime | 13.06 " | 8.29 " |
| Ferric oxide | 0.09 " | 2.50 " |
| Chlorine | 0.05 " | 1.03 " |
| Sulphuric acid | 0.26 " | 0.84 " |
| Silica | 5.13 " | 5.00 " |
| PHOSPHORIC ACID | 48.61 " | 44.48 " |
| | 100.00 per cent. | 100.00 per cent. |

Candle-nut oil is obtained by pressing the seed twice, first cold and then hot, or both times hot. The yield is 55 to 60 per cent.

The cold-drawn oil is clear, almost colorless, of an agreeable

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taste and odor, and is used as a table oil. The oil obtained by hot expression is of a brownish color and a somewhat disagreeable taste and odor. It is used for technical purposes.

The oil has a specific gravity of 1.940 at 59° F. It is quite thickly-fluid and solidifies at 32° F. It is soluble in 3 parts of ether, but almost insoluble in alcohol. It possesses strongly drying properties and is very readily saponified. Its elementary composition is:—

| | | |
|---|---|---|
| Carbon | 76.72 | per cent. |
| Hydrogen | 11.36 | " |
| Oxygen | 11.92 | " |
| | 100.00 | " |

It consists of the glycerides of linoleic and allied acids; further, stearic, palmitic, and myristic acids.

*Nitric acid* does not color the oil; *fuming nitric acid* colors it dark red-yellow, without solidification, since according to the elaïdin test all drying oils remain fluid.

*Sulphuric acid* produces an intense yellow color, which in a short time changes to a dirty syrup brown.

A mixture of *sulphuric and nitric acids* colors the oil red-brown.

*Zinc chloride* does not color the oil.

Candle-nut oil possesses strongly drying properties. Varnish prepared from it dries still more rapidly than the oil itself and more quickly than linseed-oil varnish, and as it possesses considerable hardness, is well adapted for painting.

Even when not refined the oil burns with a clear, bright flame, without carbonizing the wick to any extent, these properties being still more enhanced by refining. It yields an excellent soap, and can be used as a substitute for cocoanut oil, being preferable to linseed and hemp oils.

In the Sandwich Islands the manufacture of candle-nut oil is a considerable branch of industry, about 10,000 barrels being annually exported to Peru, Valparaiso, Mexico, California and New York. It is called by the natives *Kukui oil.*

In France the cold-drawn oil is frequently used for adulterating olive oil. Such sophistication is, however, readily detected by the specific gravity.

2. *Wood oil* or *tung oil (oleum dryandræ; Oelfirnissbaumöl*, G.; *huile de bois*, F.) from the seed of *Aleurites cordata*, Müll. = *Dryandra cordata*, Thunb. = *Elæococca vernicia*, Juss., the varnish tree or "*tung*" of the Chinese, indigenous to China and Japan, and cultivated in Algiers. By pressure the seed yields 35 per cent., and by extraction with ether, carbon disulphide, etc., 41 per cent. of oil. The cold-drawn oil is pale yellow and the warm-pressed dark brown. It is a thick oil of 0.940 specific gravity, and does not solidify on exposure to cold. Immense quantities of this oil, which possesses still more decidedly drying properties than candle-nut oil, are used in China and Japan as a natural varnish, and also for medicinal purposes.

Wood oil must, however, not be confounded with a balsam (*balsamum gurjunæ*) which is frequently called wood oil. It is obtained from *Dipterocarpus turbinatus*, Gärtn., and possesses entirely different properties. It is frequently adulterated with tung oil.

An examination of this oil by Cloëz shows it to consist of two glycerides, the ordinary olein and a peculiar glyceride, elœomargarin, the latter furnishing on saponification elœomargaric acid = margarolic acid = $C_{17}H_{30}O_2$. However, this statement does not seem probable from the formula alone, just as little as the statement that the oil on exposure to the air is converted into a fat melting only at 90° F.

About 200,000 Chinese peculs* are annually brought from Hankow in the interior of China to various Chinese markets. The oil thus far has not been exported.

3. *Elæococca verrucosa*, Juss. The oil from the seed of the tree is used in Japan for illuminating purposes.

4. *Omphalaea triandra*, Linn., indigenous to the West Indies, Guiana.

5. *Buchanania latifolia*, W. and A., a tree indigenous to the

---

* 1 pecul = 133.33 English pounds.

mountains of Coromandel and Malabar. The seed is inclosed in a stone-hard shell, tastes like pistachio nuts, and yields 40 to 50 per cent. of a very palatable table oil known as *chironji oil.* It is seldom pressed, but beside for table use is also employed for technical puposes.

6. *Mallotus Philippensis*, Müll., or *Rottlera tinctoria*, Roxb,, a tree found in Abyssinia, South Arabia, India, North Australia, etc. The fruit is covered with red glands and hair, which are brushed off and yield the coloring matter known as "*wurruss*" or "*wars*," but within the last quarter of a century introduced as a medicine under the name of "kamala."

The seed contains 20 per cent. of a clear pale-brown oil, which is known as *camul oil* and is used for many purposes.

7. *Euphorbia dracunculoides*, Lam., or *Euphorbia lanceolata*, Rottl., an annual plant of Bengal, Madras, etc. The yield of oil, which is known as "*jy-chee oil*," depends on the ripeness of the seed, and amounts sometimes to 25 per cent. It is clear, of a yellow or greenish color, and is used as lamp oil and for painting.

### *Sarmentaceæ* (*Ampelideæ*).

*Grape-seed oil* (*oleum vitis viniferæ; Traubenkernöl*, G.; *huile de raisin*, F.) from the seed of the grape, *Vitis vinifera*, Linn. The seed is separated from the marc, cleaned, dried, and finely ground, and subjected to pressure or extraction. The yield of oil varies between 10 and 20 per cent., depending very much on the quality of grapes, manner of cultivation, etc. The cold-drawn oil is of a pale-yellow color, but becomes brownish with age. When fresh it is an excellent table oil. The hot-pressed oil is of a darker, somewhat brownish color, inodorous, and has a somewhat bitter taste.

The oil extracted with ether or petroleum-ether is greenish and thick, but by filtering through charcoal is obtained bright and of a straw-yellow color. The oil dissolves readily in ether, but with great difficulty in alcohol; it saponifies with ease.

Grape-seed oil has a specific gravity of 0.9202 at 59° F. It is quite limpid, being at 59° F. only 11 times more thickly fluid than water, and at 45.5° F. only 14.2 times. It becomes thick at 5° F., and at 3° to 1.5° F. solidifies to a brownish mass of the consistency of butter.

On exposure to the air the oil remains smeary for a long time, but finally dries. It consists of the glycerides of erucic acid, stearic, palmitic, and other acids. Erucic acid, which predominates, passes, on melting with potash, into arachidic and acetic acids.

Beside as a table oil, grape-seed oil is used for illuminating purposes and in the manufacture of soap. It burns away very slow, with a bright, inodorous flame and without smoking.

### *Lineæ (Gruinales).*

Linseed oil (*oleum lini; Leinöl*, G.; *huile de lin*, F.) is obtained from the seed of *Linum usitatissimum*, Linn. The seed contains from 30 to 35 per cent. of oil, 26 per cent. of which can be obtained.

| | Fall flax. | | Spring flax. | |
|---|---|---|---|---|
| Oil | 35.20 p. c. | | 31.60 p. c. | |
| Organic substances | 53.00 " | | 57.40 " | |
| protein substances therein. | | 22.10 p. c. | | 24.00 p. c. |
| Ash | 3.15 " | | 3.20 " | |
| Water | 8.65 " | | 7.80 " | |
| | 100.00 " | | 100.00 " | |

The oil cake contains:—

| | Winter flax. | | Spring flax. | |
|---|---|---|---|---|
| Oil. | 8.63 p. c. | | 9.25 p. c. | |
| Organic substances | 73.66 " | | 72.97 " | |
| albuminous substances therein | | 27.60 p. c. | | 27 p. c. |
| Ash | 8.25 " | | 7.68 " | |
| Water | 9.46 " | | 10.10 " | |
| | 100.00 " | | 100.00 " | |

The ash is composed as follows:

| | Linseed. | Linseed cake. |
|---|---|---|
| Potash | 28.80 per cent. | 25.24 per cent. |
| Soda | 1.66 " | 1.64 " |
| Magnesia | 13.63 " | 14.40 " |
| Lime | 8.59 " | 8.45 " |
| Ferric oxide | 2.03 " | 3.52 " |
| Chlorine | 0.06 " | 1.31 " |
| Sulphuric acid | 0.10 " | 1.68 " |
| Silica | 0.40 " | 1.81 " |
| PHOSPHORIC ACID | 44.73 " | 41.98 " |
| | 100.00 per cent. | 100.00 per cent. |

For pressing, either linseed directly cultivated and ripened for that purpose is used, or the seed from the plant cultivated for the fibre; the latter seed is of unequal ripeness and does not possess the full content of oil.

The oil is obtained by either of the following three processes:—

1. *Cold-drawn oil.*—The seed is crushed, ground, or bruised and expressed without heat. This is considered the best oil, and in Russia and some parts of Germany is used as a table oil and for baking. By this process the seed yields from 20 to 21 per cent. of oil.

2. *Ordinary linseed oil.*—It is prepared in the same manner as the above, but with the assistance of heat, which gives it a disagreeable taste. The yield is from 27 to 28 per cent.; it is used for technical purposes.

3. *By extraction*, with a yield of 32 to 33 per cent.

Seed two to six months old is generally used, as the oil expressed from fresh seed is viscous and turbid.

The cake remaining after expression of the oil is known as linseed cake, and forms an important article of commerce, being extensively used as feed for cattle. Of the protein substances contained in it 87 per cent. are digestible. The nutritive value of linseed cake is nearly one and a half times as large as that of hay.

Linseed cake is frequently adulterated with considerable quantities of sand and other innutritious substances. To test it bruise a portion of the cake and mix with water. Sand, if present, will quickly deposit on the bottom. A sophistication with rape-seed cake is readily recognized by the remnants of the black-brown hulls of rape seed, those of linseed being yellowish-brown. Adulteration with bran is detected by stirring the crushed cake with water and adding solution of iodine, which, if bran be present, will give a blue coloring.

The cold-drawn oil is golden-yellow, and has a peculiar, but not disagreeable, taste and odor. The hot-pressed oil is amber-colored or brown-yellow, and has an acrid taste, due to traces of volatile fatty acids, such as butyric, valerianic, and caproic acids.

Linseed oil is somewhat thickly-fluid, and, as it belongs to the drying oils, very quickly absorbs oxygen from the air, becomes lighter in color, rancid, finally viscous, and dries. Its specific gravity is 0.9385 at 50° F., 0.9364 at 53.5° F., 0.9350 at 59° F., 0.9325 at 68° F., and 0.9300 at 77° F.

At 59° F. the oil is 9.7 times more thickly-fluid than water, and at 45.5° F. 11.5 times. At a decreasing temperature it becomes gradually thicker, pale and turbid with increasing cold, and solidifies to a solid yellow mass at —16.5° F.

At 266° F. it commences to boil, and after boiling for some time at 482° to 554° F., until it has lost about one-twelfth of its weight, it becomes thicker, viscous and sticky, and furnishes *varnish*. By heating it still further, until it has lost one-sixth of its weight, it becomes still thicker, the product being *printers' ink*.

By heating linseed oil to 608° to 707° F. it ignites and burns quietly, without further external heating, until tar and carbon remain. By interrupting the burning by covering the boiler, there remains a brown, turpentine-like substance, the so-called *bird-lime*.

Linseed oil is soluble in 1.5 parts of ether, in 40 parts of 90 per cent. spirit of wine, in 5 parts of boiling 90 per cent. spirit of wine, and in 5 parts of absolute alcohol. It is miscible in all proportions with chloroform, carbon disulphide, oil of turpentine, etc.

Its elementary composition is:—

| | Cold drawn. | Hot pressed. |
|---|---|---|
| Carbon | 78.11 p. c. | 75.27 p. c. |
| Hydrogen | 10.96 " | 10.88 " |
| Oxygen | 10.93 " | 13.85 " |
| | 100.00 " | 100.00 " |

Linseed oil is a mixture of linolein, the glyceride of linoleic acid, which forms the principal constituent—about 80 per cent. —olein, palmitin, and myristin.

The products of saponification are also glycerin, linoleic acid, oleic acid, or an allied acid which by dry distillation gives sebacic acid, palmitic and myristic acids.

That linoleic acid is not a unitary chemical body has been plainly shown by Hazura's investigations referred to, Vol. I, p. 164. The practical result of the investigation is as follows:

Since the drying of oils consists in the absorption of oxygen with the formation of solid products of oxidation, and since all oils whose fluid fatty acids consist only of oleic acids $C_{18}H_{34}O_2$ belong to the non-drying oils, it may be supposed that an oil dries the more incompletely, the more oleic acid it contains. Further there can be no doubt that linolenic and isolinolenic acids $C_{18}H_{30}O_2(OH)_6$, which contain 6 free valencies, are capable of absorbing more oxygen than linolic acid $C_{18}H_{32}O_2$, which contains four free valencies. Hence, an oil will dry the better the greater the preponderance of the acids $C_{18}H_{30}O_2(OH)_6$ is.

The correctness of the latter statement is confirmed by the appended table of Fox, according to which linseed oils of different origin are capable of absorbing varying quantities of oxygen.

According to Fox the capacity of various oils for absorbing oxygen is as follows:

| 1 gramme of | absorbs oxygen | |
|---|---|---|
| Linseed oil, Baltic | 191.0 | cubic centimeters. |
| " Black Sea | 186.0 | " |
| " Calcutta | 126.0 | " |
| " Bombay | 130.0 | " |
| " American | 150.0 | " |
| Cottonseed oil, refined | 24.6 | " |
| Rapeseed oil, brown | 20.0 | " |
| Colza oil | 17.6 | " |
| Olive oil, best quality | 8.7 | " |
| " refined | 8.2 | " |

If linseed oil be saponified with lead oxide, the resulting lead oleate dissolved in ether, and the ethereal solution applied to glass-bells, there remains, after the evaporation of the ether, a dry, hard, white residue—oxylinoleic acid—which is a product of oxidation of linoleic acid. As shown by the investigations of Hazura, this product is not a unitary body. Mulder already distinguished two modifications of oxylinoleic acid—a white and a red—which can be converted one into the other. By treating linseed oil with nitric acid a dark-red viscous mass is formed, which is identical with the red oxylinoleic acid. By long exposure to the air the colorless oxylinoleic acid suffers, when frequently sprinkled with ether, a further alteration; it loses its sticky nature and acid property, and is converted into a neutral body—linoxyn, $C_{32}H_{14}O$—insoluble in ether, which is the most important basis of linseed oil paints, and very likely is the product of most drying oils.

In boiled linseed oil linoxyn is formed within a few days. It is an amorphous, elastic, non-hygroscopic body, which is heavier than water and insoluble in it, as well as in alcohol and ether. In a mixture of chloroform and alcohol, or carbon disulphide and oil of turpentine, it swells up, and finally dissolves.

When exposed in a thin layer to the air, linseed oil gradually dries to a transparent, resinous, more or less elastic mass, increasing thereby in weight in consequence of the absorption of oxygen, while carbonic acid, formic acid, acetic acid and water formed from the glycerin by the absorption of oxygen are at the same time yielded up.

If linseed oil is kept for a length of time in half-filled bottles, it becomes more thickly-fluid, and does not dry as well as formerly. By this treatment it acquires, however, the property of dissolving with greater ease in alcohol, and is in this state added to several spirit varnishes to decrease their brittleness.

Thoroughly dried linseed oil is completely deprived of linolein, free linoleic acid, olein, palmitin or myristicin, and contains only free fatty acids and linoxyn. All the glycerides are decomposed; what remains behind is linoxyn mixed with palmitinmyristin and oleic acid, the latter being oxidized after drying for some time.

Linseed oil possesses the property of dissolving not inconsiderable quantities of sulphur; thus there are dissolved at

77° F. = 0.630 per cent. sulphur.
140° " = 1.852 " "
203° " = 2.587 " "
266° " = 4.935 " "
320° " = 9.129 " "

On cooling, the sulphur separates very slowly. If, however, linseed oil be heated to boiling and up to ¼ per cent. of its weight of sulphur be added, in small portions and with constant stirring, a dark-brown viscous mass is formed, while sulphuretted hydrogen, carburetted hydrogen and other products of decomposition are evolved. This mass constitutes the *balsamum sulphuris*. In thin layers it is red-brown and perfectly transparent, and of an offensive odor; it is insoluble in water and alcohol, but readily soluble in ether and fat oils.

The property of absorbing oxygen and consequent drying is materially increased by boiling the linseed oil by itself or with certain substances such as lead oxide, minium, borate of manganese, etc., whereby linseed oil varnishes are obtained. The absorption of oxygen may, however, be also increased by metals in a finely-divided state.

Livache has investigated the action of finely-divided lead, copper and tin upon oils; lead obtained by precipitating lead solution by means of zinc proving most effective. By moistening such lead with oil and exposing it to the air it increases in weight the more rapidly the more readily the oil dries under ordinary conditions. The increase in weight of various oils with or without lead amounts to:

| | With lead | | Without lead. |
|---|---|---|---|
| | in two days. | in seven days. | in eight months. |
| Linseed oil............. | 14.3 per cent. | — | 11.0 per cent. |
| Nut oil................. | 7.9 " | — | 6.0 " |
| Poppy-seed oil.......... | 6.8 " | — | 3.7 " |
| Cotton-seed oil......... | 5.9 " | — | 0.8 " |
| Beechnut oil............ | 4.3 " | — | 2.6 " |
| Colza oil............... | — | 2.9 per cent. | 2.6 " |
| Sesamé oil.............. | — | 2.4 " | 2.0 " |
| Peanut oil.............. | — | 1.8 " | 1.3 " |
| Rubsen oil.............. | — | 2.9 " | 0.8 " |
| Olive oil............... | — | 1.7 " | 0.7 " |

The peculiar behavior of cottonseed oil explains its use for adulterating linseed oil as well as olive oil.

*Nitric acid* of 1.30 specific gravity colors linseed oil sulphur-yellow, and if the acid contains some nitrous acid, yellowish-greenish, which, however, soon changes to red-brownish. *Fuming nitric acid* produces on the point of contact a red-brown zone, while the acid itself is colored green. By shaking, the mixture becomes dirty cherry-brown, which later on becomes somewhat paler.

*Nitric acid* of 1.4 specific gravity mixed with the same weight of oil (about 6 grammes of each) gives, after shaking for about 30 minutes at 61° F., the following reactions:

| | Upper layer. | Lower layer. |
|---|---|---|
| Pure crude oil. | light cinnamon brown. | colorless. |
| Crude oil with 5 p. c. rosin oil. | " " | straw-color. |
| " " 12 " " | dark olive. | deep straw-color. |
| " " 50 " " | blackish. | light orange. |
| Pure boiled oil. | cinnamon brown. | colorless. |
| Boiled oil with 5 p. c. rosin oil. | " | straw-color. |
| " " 12 " " | dark olive. | deep straw-color. |
| " " 50 " " | blackish. | light orange. |

*Sulphuric acid* causes first a green color, which, however, soon changes to brown. By mixing 10 drops of linseed oil with 3 drops of sulphuric acid a red-brown resinous mass is formed. A mixture of sulphuric and nitric acids produces first a yellow color, which changes to gray-green and, later on, to a dirty green-brown.

Five parts by weight of *concentrated sulphuric acid*, added to 15 parts by weight of linseed oil, cause the temperature to increase from 59° F. to 194° F., a difference of 135°.

The temperature of linseed oil adulterated with colza oil will rise only:—

| | | | | | | |
|---|---|---|---|---|---|---|
| With | 5 | per cent. | of colza | oil to | 163.5° | F. |
| " | 10 | " | " | " | 160 | " |
| " | 15 | " | " | " | 157 | " |
| " | 20 | " | " | " | 153.5 | " |
| " | 25 | " | " | " | 150 | " |
| " | 30 | " | " | " | 146.5 | " |

Solution of *nitrate of silver* causes a somewhat red-brown coloring.

Of *iodine* up to 15 per cent. is dissolved by linseed oil.

*Zinc chloride* produces no effect.

*Ammonia, potash lye, and lime-water,* produce yellowish liniments.

*Caustic alkalies* saponify linseed oil very quickly, yielding soaps of little solidity. By saponification linseed oil gives 95 per cent. of fatty acids and 5 per cent. of glycerin.

*The detection of linoleic acid in other oil mixtures* is, according to R. Hazura, readily accomplished if such admixture amounts to at least 1 per cent. For establishing the presence of linoleic acid in olein such method is of special value because, if olein mixed with linoleic acid is used in wool spinning, it causes considerable inconvenience to the operators.

Saponify 50 grammes of the olein to be tested with dilute alcohol in a water bath, free the resulting soap from alcohol, and dissolve it in 1 quart of water. The solution, which should be strongly alkaline, is then mixed, slowly and with constant stirring, with 1 quart of a 5 per cent. solution of potassium permanganate. After ½ to 1 hour the fluid is filtered off from the manganic hydrate separated, the filtrate acidulated with sulphuric acid and again filtered off from the precipitate formed. The filtrate thus obtained is neutralized with caustic potash, evaporated to about 300 cubic centimeters and again acidulated with sulphuric acid, whereby another precipitate is formed. Without removing the precipitate, the acid fluid is now shaken with ether. If the precipitate is completely dissolved it consists of azelaic acid $C_9H_{16}O_4$, and the olein in this case is free from linoleic acid.

If the precipitate is not dissolved by the ether, the presence of linoleic acid may be presumed. However, to be entirely sure, the precipitate insoluble in ether is filtered off, several times recrystallized from water or alcohol, with the addition of animal charcoal, and, when air-dry, its fusing point is determined. If the latter lies above 320° F., linoleic acid is present without doubt.

If a larger quantity of products of oxidation fusing at above 320° F. has been obtained, the acid number may be determined as a further proof; it should not exceed 150, since the acid number of the hexoxystearic acids contained in this product of oxidation is 147.3.

The above-mentioned formation of a brown resinous mass by mixing 10 drops of linseed oil with 3 drops of sulphuric acid is very characteristic as regards adulterations.

In the presence of foreign oils, such as colza, poppy-seed, nut, cotton-seed oils, etc., the oil does not form a brown, viscous, resinous mass. The linseed oil alone is changed to a resin, the brown resinous flakes floating in the admixed oils.

An admixture of fish oils is readily recognized by their peculiar odor and taste, or by phosphoric acid of the consistency of syrup, which colors linseed oil yellow-brown, but fish oils red, which is, however, soon changed to black-brown. Even Scotch cod-liver oil, which is now brought into commerce almost inodorous and of a very pale color, shows this change with phosphoric acid.

An adulteration with even $\frac{1}{10}$ per cent. of fish oil can also be detected by the red color produced by sulphuric acid.

*Concentrated hydrochloric acid* added to linseed oil gives a yellow mixture. If hemp oil be present the mixture is green-yellow, because hydrochloric acid mixed with hemp oil gives a characteristic grass-green color.

For testing linseed oil as to the presence of mineral oil, Finkener gives the following method:

Pour into a glass cylinder of about 0.7 inch internal diameter and 7.87 inches high a layer of oil 1.57 inches deep, and upon it a layer of aniline aboute 5.11 inches deep, so that the cylinder contains a total quantity of fluid 6.69 inches deep. After vigorously shaking the contents let the cylinder stand in the temperature of a cellar for 24 hours. Pure linseed oil or pure boiled linseed oil remains clear, while in the presence of mineral oil an oily layer separates on the surface, which is readily recognized by slightly moving the fluid.

An addition of resins (colophony, etc.) is recognized by the white lumpy precipitate which is formed by shaking the linseed oil with spirit of wine, and compounding the alcoholic solution with alcoholic solution of sugar of lead.

For the same purpose Finkener shakes one drop of the oil with 1 cubic centimeter of acetic acid, and then adds one drop of concentrated sulphuric acid. In the presence of resin an intensely purple-red color appears, which in a short time disappears.

| | |
|---|---|
| Saponification number of linseed oil | 264.7 |
| Iodine number of linseed oil | 177–178 |
| " " of the fatty acids | 155 |
| Fusing point of the fatty acids | 62.6° F. |
| Solidifying point of the fatty acids | 56.3° F. |

Linseed oil is extensively used in the manufacture of paints, printing inks, varnishes, etc,, and for the preparation of water-proof stuff, floor cloths, elastic rollers, etc. Linseed oil boiled to the consistency of caoutchouc served formerly for the manufacture of bougies, catheters, and elastic probes.

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To prepare such caoutchouc-mass the linseed oil is brought to a very thick consistency by boiling for at least twenty-four hours, and is then boiled a few hours longer with dilute nitric acid. The mass is plastic and becomes hard in contact with the air. By dipping in hot water it regains its softness, and on cooling solidifies again to a caoutchouc-like mass.

Linseed oil is sold under four different forms, viz., raw, refined, boiled, and artists' oil. Of these by far the most important, commercially speaking, is the boiled.

The selection of the oil to be boiled is a matter of the utmost consequence to the oil boiler and varnish maker, since the beauty and durability of the paints mixed with it depend largely upon its quality. If the seed be not fully ripe, the oil expressed abounds in mucilage, water and pulpy matter, while more or less acid is also present. If on the other hand the seed be full grown and ripe, the oil is limpid, pale colored, and of a highly refractive power.

To summarize the chemical changes induced in linseed oil by exposure to the air, it may be said that the oil dries, or, in other words, that it passes, by mere absorption of oxygen from the atmosphere, from a fluid into a solid state. From this known property was derived the theory that it was necessary to heat with certain metallic oxides, from which the oil might derive oxygen previous to its exposure to the air, thus shortening the time subsequently needed for complete absorption. Various oxides were made use of for this purpose—plumbic oxide (litharge), red lead, and anhydrous binoxide of manganese. More recently the two last-named oxides have been employed conjointly, and to these agents have been added lead oxide of plumbic-acetate, zinc sulphate, etc. The occasional addition of umber serves only to deepen the color of the oil.

The reasons for boiling linseed oil are principally two. First, that drying may be facilitated when the oil is spread upon thin surfaces, either alone or mixed with paint; and second, that it may serve as a vehicle for the mechanical suspension of the finely-divided particles constituting a paint, thus enabling them to adhere to the surface on which they are spread. It must not run into drops, nor must it leave the coloring matter behind; the color must be carried by it, evenly diffused, over the whole surface over which the paint is laid. The object to be obtained is to secure a coating impervious alike to liquids and gases.

It may sometimes happen that even when the process of boiling has been performed properly and with the utmost care, the result may prove unsatisfactory to the consumer. This may sometimes be traced to circumstances entirely independent of the process of manufacture; the quality of the seed—green or ripe, new or old—the climate, and the soil in which the seed was grown, all these exert a marked influence upon the product. The different varieties of seed call for separate and distinctive treatment.

All oil boilers should test the drying qualities of each batch of oil. One tried and approved test is to dip into the oil, when cool, a piece of well-sized paper, which is afterwards hung up to dry. Thoroughly well-boiled oil will produce a crystalline surface over nearly the entire portion of the sheet dipped in the oil. If the boiling has been imperfect the upper portion of the paper's surface will be simply greasy, and only the lower portion will show the varnished coating. When the touch of a finger to the submerged portion of the paper shows no grease adhering to it, the boiling may be said to be complete.

Reference has already been made to the use of heat for the purpose of driving off the mucilage from the seed. The philosophy of the process consists in the throwing off of the water (probably held by the mucilage), a yellow froth mounting to the surface of the gradually heating oil. The continued application of heat evolves numerous gases. Before the attainment of the result hoped for from the heat, however, a temperature of between 600° and 700° F. has been obtained. When the partially destructive distillation has effected the desired change in the oil, the liquid assumes a deep, permanent (sometimes reddish) brown. In the opinion of some manufacturers the depth of this brown is the best test by which to gauge the thoroughness of the boiling.

The old-fashioned way of fire heating has been to a very considerable extent abandoned, steam being employed as a substitute.

Linseed oil is also used in the manufacture of soap and as a lamp oil. For the latter it is not well adapted, as it forms much soot in burning.

The principal market for flax-seed and linseed oil is England, flax-seed being imported from all parts of the world and linseed oil exported.

According to the Extra Census Bulletin, No. 67, Washington, March 15, 1894, there were in the United States 62 establishments engaged in the manufacture of linseed oil, the total value of their products amounting to $23,534,306.

2. ***Linum strictum***, Linn., indigenous to the Punjab and Thibet, where it is known as "*Basant*," "*Bub-basant*." In Afghanistan it is cultivated as a forage plant and for pressing oil, but not as a textile plant. The seed yields an oil closely resembling linseed oil, which is employed for the same purposes as the latter.

### *Cucurbitaceæ* (*Peponiferæ*).

1. ***Pumpkin-seed oil*** (***oleum peponis***, ***Kürbiskernöl***, G.; ***huile de pepins de citrouille***, F.) from the seed of ***Cucurbita pepo***, Linn., the common pumpkin. The oil, which is generally expressed from the peeled seed, is clear, pale yellow or almost colorless, inodorous, and of an agreeable sweetish taste. Next to castor and olive oils it is the most thickly-fluid oil, being 20.5 times more thickly fluid than water at 59° F., and 26.5 times more at 45.5° F. It dries very slowly in the air, has a specific gravity of 0.9231 at 59° F., and solidifies to a gray-yellow mass at 5° F.

Subjected to the elaïdin test the oil becomes somewhat thicker only after 24 to 48 hours, the color passing thereby into dark brown-red.

*Sulphuric acid* of 1.828 specific gravity produces on the line of contact with the oil a pale-green color, and when mixed with the oil a dark-brown color.

*Sulphuric acid* of 1.530 specific gravity, when mixed with the oil, produces a dirty-green, quite dark emulsion.

*Alcoholic silver solution* produces after three hours no change of color, a partial reduction appearing only after 12 hours.

| | |
|---|---|
| Saponification number of the oil | 188.1 |
| Iodine number of the oil | 121 |
| Fusing point of the fatty acids | 82.4° F. |
| Solidifying point of the fatty acids | 76° F. |

The oil pressed from shelled seed is used for edible and culinary purposes, inferior qualities for lamp oil.

The oil extracted from the comminuted seed by means of ether is used as a remedy for tape worm.

2. *Watermelon-seed oil* (*oleum citrulli; Wassermelonenöl*, G.; *huile de melon d'eau*, F.) from the seeds of *Cucumis citrullus*, Linn. The seed is brought from Senegal to France, where it is pressed; it yields as much as 30 per cent. of oil, which is very limpid and pale yellow. It is used as a table oil, and in the manufacture of soap.

3. *Cucumber-seed oil* (*oleum cucumeris; Gurkenkernöl*, G.; *huile de concombre*, F.) from the seeds of *Cucumis sativus*, Linn., the common cucumber. The seed contains 25 per cent. or more of a pale yellow oil, which is used for the same purpose as the preceding.

4. *Melon-seed oil* (*oleum melonis; Melonenöl*, G.; *huile de melon*, F.). The seed of *cucumis melo*, Linn., forms quite an important article of commerce on the coast of West Africa. In Gambia it serves as food and is also partially sold, chiefly to French merchants. It contains 30 to 32 per cent. of oil. The latter is pale yellow and odorless and has a sweetish taste. It dries very slowly and is chiefly used as a table oil.

5. *Cucumis colocynthis*, Linn., = *Citrullus colocynthis*, Sohead, the bitter apple, indigenous to Asia and North Africa. The seed yields an oil known as *colocynth oil*.

6. *Lagenaria vulgaris* D. C. =*Cucurbita lagenaria* Linn., the gourd.

7. *Luffa acutangula*, Roxb.

8. *Feuillea cordifolia*, Vell, a climbing plant of Brazil. The seed contains 32 to 33 per cent. of a clear, inodorous, yellow oil of mild taste. It has a specific gravity of 0.9309 at 59° F.

9. *Koëme oil* (*Castanhasöl*, G.; *huile de noix d'Inhambane* F.) from the seed of *Telfairia pedata*, Hook, indigenous to southeastern Africa. The ripe fruit is three feet long and 8 to 10 inches in diameter. The seed is the size of a chestnut, one fruit containing up to 200; and it has a very agreeable almond-like taste. Two hundred pounds of seed give only 35 lbs. of oil, which is equal to the finest olive oil. This small yield is no doubt due to crude treatment, since the seed is claimed to contain 33 per cent. of oil.

10. *Telfairia occidentalis* is indigenous to West Africa, where it is also cultivated. The oil obtained from the seed is sweet and mild and is used as table oil.

### *Cruciferæ* (*Rhoeadeæ*).

1. *Camelina oil* or *German sesamé oil* (*oleum camelinæ; Leindotteröl*, G.; *huile de cameline*, F.) is contained in the seed of *Myagrum sativum*, Linn. = *Camelina sativa*, Crz., and in that of *Myagrum dentatum*, plants indigenous to Asia and Europe. The seed contains 25 to 30 per cent., yielding by cold pressure 18 to 20 per cent., by hot pressure 23 to 25 per cent., and by extraction up to 28 per cent.

| | Seed. | | Oil-cake. | |
|---|---|---|---|---|
| Oil | 29.50 p. c. | | 6.97 p. c. | |
| Organic substance | 56.58 " | | 73.03 " | |
| protein substances therein | | 25.30 p. c. | | 31.42 p. c. |
| Ash | 6.42 " | | 8.85 " | |
| Water | 7.50 " | | 11.15 " | |
| | 100.00 " | | 100.00 " | |

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The oil cake is an excellent feed, especially for fowls.

The ash of the seed contains:

| | | |
|---|---|---|
| Potash | 13.30 | per cent. |
| Soda | 5.60 | " |
| Magnesia | 3.20 | " |
| Lime | 21.00 | " |
| Ferric oxide | 1.40 | " |
| Chlorine | 4.20 | " |
| Sulphuric acid | 4.20 | " |
| Silica | 7.00 | " |
| PHOSPHORIC ACID | 40.10 | " |
| | 100.00 | " |

The cold-drawn oil is of a somewhat lighter color than the hot-pressed, both having a golden-yellow color and peculiar acrid odor and taste, the latter somewhat bitter; this, however, diminishes by storing.

The oil has a specific gravity of 0.9228 at 59° F., and at the same temperature is 13.1 times more thickly-fluid than water. It becomes very thick at 5° F., and solidifies to a white butter at —0.5° to —2° F. Alcohol takes up somewhat more than 1 per cent. of oil.

The oil dries very slowly in the air, and boiled with litharge or borate of manganese gives a very slowly drying varnish. After saponification it forms a soft soap. It burns with a bright flame, and, if carefully prepared, without soot. It consists of glycerides of oleic and palmitic acids and some erucic acid and an acid related to linoleic acid. Its elementary composition is:

| | | |
|---|---|---|
| Carbon | 77.12 | per cent. |
| Hydrogen | 11.95 | " |
| Oxygen | 10.93 | " |
| | 100.00 | " |

*Nitric acid* containing some nitrous acid colors both the crude and refined oil brick red, while fuming nitric acid colors them dirty brown-red.

*Sulphuric acid* dropped into the oil produces first a yellow color with bluish streaks, which later on changes to orange and then into gray-brownish. The oil becomes thick with sulphuric acid.

A mixture of *sulphuric* and *nitric acids* effects a brownish-red coloring of the oil.

*Zinc chloride* produces a greenish color.

*Solution of nitrate of silver* is colored black.

The cold-drawn oil, after settling, is occasionally used as a table oil, but generally as a lamp oil, and principally in the manufacture of soap.

2. *Cassweed-seed oil* (*Täschelkrautsamenöl*, G.; *huile de Thlaspi*, F.), from the seed of *Thlaspi arvense*, Linn., a weed found in every part of Europe. The seed contains 20 per cent. of oil, which is pressed in France and used as a lamp oil.

3. *Cress-seed oil* (*Gartenkressensamenöl*, G.; *huile de cresson alénois*, F.), from the seed of *Lepidium sativum*, Linn. The seed contains 50 to 60 per cent. of a fat oil resembling black mustard-seed oil, and by distillation yields also a volatile oil. The fat oil has a brownish-yellow color and a peculiar, not agreeable, odor and taste. Its specific gravity is 0.924 at 59° F., and at the same temperature it is 11.4 times more thickly-fluid than water. It becomes turbid between 20.75° and 14° F., and solidifies to a yellow mass at 5° F. It dries slowly. It is used as a lamp oil and in the manufacture of soap.

4. *Hesperis oil* (*Rothrepsöl*, G.; *huile de julienne*, F.) is obtained from the seed of *Hesperis matronalis*, a plant indigenous to Middle and Southern Europe, and also cultivated in gardens. The seed contains 25 to 30 per cent. of oil, which, when fresh, is of a greenish color, changing, however, to brownish by age. It is almost inodorous, and has a somewhat bitter taste. Its specific gravity is 0.9282 at 59° F., and at this temperature it is 9.8 times more thickly-fluid than water. At 5° F. it commences to thicken, and solidifies at —7.6° to —9.4° F. It dries quickly, but is only used as lamp oil.

### *Papaveraceæ* (*Rhoeadeæ*).

1. *Poppy-seed oil* (*oleum papaveris; Mohnöl*, G.; *huile d'oeillete, huile de pavot du pays*, F.), from the seed of *Papaver somniferum*, Linn., the opium poppy. Two varieties are cultivated, *Papaver album* and *Papaver nigrum*, the first bearing white seeds, which are principally used for medicinal purposes, and yield a finer oil than *Papaver nigrum*, with bluish-black seeds. The black variety, the cultivation of which yields a better return, is principally used for obtaining oil. The percentage of oil is the same in both varieties, varying between 50 and 60 per cent.

| | White poppy seed. | | Black poppy seed. | |
|---|---|---|---|---|
| Oil | 55.62 p. c. | | 51.36 p. c. | |
| Organic substances, | 32.11 " | | 35.14 " | |
| protein substances therein. | | 16.89. p. c. | | 17.50 p. c. |
| Ash | 3.42 " | | 4.00 " | |
| Water | 8.85 " | | 9.50 " | |
| | 100.00 " | | 100.00 " | |

### *Poppy seed cake.*

| | White poppy. | | Black poppy. | |
|---|---|---|---|---|
| Oil | 8.43 p. c. | | 8.20 p. c. | |
| Organic substance | 71.97 " | | 71.34 " | |
| nitrogenous bodies therein | | 31.35 p. c. | | 32.40 p. c. |
| Ash | 11.10 " | | 10.86 " | |
| Water | 8.50 " | | 9.60 " | |
| | 100.00 p. c. | | 100.00 | |

The ash contains:

| | | |
|---|---|---|
| Potash | 13.62 per cent. | 11.05 per cent. |
| Soda | 1.03 " | 2.50 " |
| Magnesia | 9.49 " | 5.00 " |
| Lime | 34.35 " | 35.00 " |
| Ferric oxide | 0.43 " | 3.40 " |
| Chlorine | 4.58 " | 5.15 " |
| Sulphuric acid | 1.92 " | 2.45 " |
| Silica | 3.22 " | 4.00 " |
| PHOSPHORIC ACID | 31.36 " | 31.45 " |
| | 100.00 per cent. | 100.00 per cent. |

By cold and warm pressure two qualities of oil are obtained. The yield varies between 47 and 55 per cent. The first pressure yields 33 to 40 per cent. of quality I., and the second warm pressure 22 to 25 per cent. of quality II. The first pressure gives the white oil and the second pressure the red oil, which can also be directly obtained by heating seed of poor quality. It is only used in the manufacture of soap, *i. e.*, as an addition in the preparation of hard soap.

Cold-drawn poppy-seed oil is of a pale yellow color, clear, limpid, and has a slight, peculiar odor. It is used as a table oil, many preferring it to olive oil, for the adulteration of which it is extensively used.

The second quality, obtained by hot pressure, has an acrid taste and a strong odor.

Poppy-seed oil dries and keeps well. Its specific gravity is 0.9250 at 59° F., and at that temperature it is 13.6 times more thickly-fluid than water. It does not freeze readily, remaining clear and thickish at 5° F., and solidifying to a thick white mass only at —4° F. It is soluble in equal volumes of ether, in 25 parts of cold and in 6 parts of boiling alcohol.

It dries somewhat quicker than linseed oil, but is only used for fine painting.

As regards its chemical composition, it consists principally of linolein and glycerides of oleic, stearic, and palmitic acids. Myristic and lauric acids are wanting.

Its elementary composition is:—

| | | |
|---|---|---|
| Carbon | 76.63 | per cent. |
| Hydrogen | 11.63 | " |
| Oxygen | 11.74 | " |
| | 100.00 | " |

*Nitric acid* has no effect upon poppy-seed oil; in the presence of some *nitrous acid* it becomes yellowish red; by testing with *fuming nitric acid* the reddish shade becomes somewhat more pronounced.

*Sulphuric acid* of 1.70 specific gravity gives first a grayish mixture with a slightly greenish tinge, which changes to a dirty yellowish to dirty brownish color. Sulphuric acid of 1.84 specific gravity gives first a pure yellow and then a greenish-yellowish color, which quickly changes to brownish.

A mixture of *sulphuric and nitric acids* gives a pale greenish-yellowish color, which is, however, very transitory, changing to a somewhat brick red, and then to a dirty brownish color.

The temperature of a mixture of 15 grammes of poppy-seed oil and 5 grammes of concentrated sulphuric acid rises 126° F., *i. e.*, from 59° F. to 185° F.

*Ammonia* gives with poppy-seed oil a white emulsion, and potash lye a slightly yellowish one.

*Concentrated hydrochloric acid* and an equal volume of poppy-seed oil, shaken frequently with a little cane sugar, are colored light brown.

Poppy-seed oil, when heated to boiling, becomes covered with a quantity of froth, which remains standing for some time. Olive oil does not show this peculiarity, which, therefore, may serve for distinguishing the two oils.

| | |
|---|---|
| Saponification number of the oil | 193–194 |
| Iodine number of the oil | 134–135 |
| Fusing point of the fatty acids | 69° F. |
| Solidifying point of the fatty acids | 62° F. |

Poppy-seed oil is readily saponified, yielding a hard soap which does not change in the air.

The simplest test of poppy-seed oil is by taste and odor, and allowing a few drops to dry upon a glass plate in a water-bath or in a warm place. The residue should be clear and hard, and not viscous.

2. *Horned poppy-seed oil (oleum glaucii; Hornmohnöl*, G.; *huile de pavot cornu*, F.) is obtained from the seed of *Glaucium luteum*, Scop., the yellow-horn poppy. The oil, of which the seed contains 30 to 35 per cent., is inodorous and tasteless, of a pale yellow color, and has a specific gravity of 0.913 at 59° F. It is used as a table and lamp oil, in the manufacture of soap, and for grinding oil colors.

3. *Shial-Kanta oil (Argemoneöl*, G.; *huile de pavot epineux*, F.) from the seeds of *Argemone Mexicana*, Linn., the spiny poppy, a plant resembling the poppy, and indigenous to America and the East Indies. The seed contains 25 to 30 per cent. of oil of an orange color and without taste or odor. Its specific gravity is 0.919 at 59° F. It is used as a lamp oil and lubricant. In the West Indies and Mexico it is employed as a remedy for headache and as a purgative.

### *Resedaceæ (Rhœadeæ).*

*Weld-seed oil (oleum resedæ luteolæ; Wausamenöl*, G.; *huile de gaude*, F.) from *Reseda luteola*, Linn., the weld, which is cultivated in many parts of Europe on account of the yellow coloring matter contained in all its parts. The seed contains 20 to 30 per cent. of dark greenish oil of a bitter taste and disagreeable odor. It is very limpid, has a specific gravity of 0.9058 at 59° F., and solidifies only at —22° F. It dries quickly in the air, and is used as a lamp oil and in the manufacture of varnish.

No animal will eat weld, and for that reason the oil cake is of no value for feed, and can only be used as fuel.

### *Solaneæ (Tubifloræ).*

1. *Belladonna-seed oil (oleum belladonnæ; Tollkirschensamenöl*, G.; *huile de belladonne*, F.), from *Atropa belladonna*, Linn., the deadly nightshade. In Wuertemberg the seed is collected and pressed, the narcotic constituents remaining in the cake. The oil is clear, golden-yellow, of a mild taste, non-poisonous, and somewhat thicker than linseed oil. Its specific gravity is 0.925 at 59° F., at 3° F. it becomes thick, and solidifies at —16.5° F. It dries very slowly. It is used as a table oil, lamp oil, and lubricant.

2. *Tobacco-seed oil (Tabaksamenöl*, G.; *huile de tabac*, F.), from *Nicotiana tabacum*, Linn., the tobacco plant. The oil, of which the seed contains from 30 to 32 per cent., is pale green-yellow and of the consistency of hemp oil. It is inodorous

and of a mild taste, and does not possess the acridity and odor of tobacco. Its specific gravity is 0.9232 at 59° F., it becomes thick at 5° F., and solidifies at —13° F. It dries quickly, and is used as lamp oil.

### *Labiatæ.*

*Lallemantia oil*, from the seed of *Dracolephalum aristatum*, Berth., or *Lallemantia iberica*. This plant, indigenous to Tauris and the Caucasus, has been recommended as an oil-plant. It is cultivated in the neighborhood of Kief. It has three-cornered chocolate-brown seeds slightly curved to one side. They contain 23.79 per cent. nitrogenous substance, 3.96 per cent. ash, and 33.52 per cent. oil. The oil possesses great drying properties, surpassing in this respect, according to Richter, even linseed oil. A sample in a watch crystal was covered in 9 days with a thick resinous skin. 10 grammes of oil of 64.4° F. became heated to 248° F. by the addition of 2 grammes of concentrated sulphuric acid.

With the elaïdin test (10 grammes oil, 5 grammes nitric acid, and 1 gramme mercury) a dark red pasty mass is separated after shaking three minutes.

Specific gravity of the oil at 68° F., 0.9336; solidifying point —31° F.

| | |
|---|---|
| Saponification number of the oil | 185 |
| Iodine number of the oil | 162.1 |
| Iodine number of the fatty acids | 166.7 |
| Fusing point of the fatty acids | 72° F. |
| Solidifying point of the fatty acids | 51.8° F. |

### *Synanthereæ* (*Compositæ*).

1. *Sunflower-seed oil* (*oleum helianthi annui; Sonnenblumenöl*, G.; *huile de tournesol*, F.) from the seed of *Helianthus annuus*, the common sunflower. The great varieties of valuable properties belonging to the sunflower seed have been much neglected in this country. While the plant is largely and profitably cultivated in Germany, Hungary, and Russia, it is here raised only as an ornamental plant by a few country housewives,

or either for fattening poultry or on account of its supposed value in warding off malaria. But no plant produces such fine honey and wax, and when the flower is in blossom bees abound in it. The produce will be according to the nature of the soil and mode of cultivation; but the average has been found 50 bushels of the seed per acre, which will yield 50 gallons of oil The marc or refuse of the seed of the above quantity, after the oil has been expressed, made into cake, will produce 1,500 pounds. But, besides, every portion of the plant can be utilized. The leaves furnish an excellent fodder, the stalks are prized as fuel, and their ashes, which contain 10 per cent. of potash, are readily sold to soap-makers. The seed should be planted about six inches apart and about one inch deep. When the plant is about one foot high it is earthed up, and will then require no further attention. Every single seed will produce a thousand or more. On some grounds two crops may be grown at the same time. When the farmer has given his early potatoes a last hoeing, he may plant this seed twelve inches apart in the ridges.

In Russia, where the cultivation of the plant and manufacture of the oil are carried on on a large scale, the *grandiflora* is the variety grown. It grows in one slender stalk five feet high, producing one monstrous head. A good, warm summer is required to bring the flower to perfection. The seed is threshed out usually by flails. It is then thoroughly dried and hulled. To accomplish this, specially-constructed mills or grindstones are used, a fan is commonly attached, and the motive power is a pair of horses. That the manufacture in Russia is found to be a source of profit is shown by the fact that sunflower cultivation is constantly increasing. The same good results could, no doubt, be attained in some of the Northwestern States, Minnesota, Wisconsin, etc., the climate of which resembles that of Southwestern Russia, where the cultivation of the plant is extensively conducted.

The hulled seed contains:—

| | German seed. | | Russian seed. | |
|---|---|---|---|---|
| Oil | 33.48 p. c. | | 34.25 p. c. | |
| Organic substances | 54.04 " | | 54.39 " | |
| protein substances therein | | 14.12 p. c. | | 18.80 p. c. |
| Ash | 2.86 " | | 3.56 " | |
| Water | 9.62 " | | 7.80 " | |
| | 100.00 " | | 100.00 " | |

The produce is 28 to 30 per cent., 18 to 20 per cent. being obtained by the first cold pressure and the remainder by warm pressure.

By extracting the seed with petroleum-ether, 26 to 27 per cent. of oil may be obtained.

The oil-cake contains:

| | | |
|---|---|---|
| Oil | 6.60 per cent. | |
| Organic substances | 75.03 " | |
| nitrogen therein | | 37.30 per cent. |
| Ash | 8.13 " | |
| Water | 10.24 " | |
| | 100.00 " | |

The ash of the seed contains:

| | |
|---|---|
| Potash | 16.23 per cent. |
| Soda | 7.41 " |
| Magnesia | 12.29 " |
| Lime | 7.63 " |
| Ferric oxide | 1.60 " |
| Chlorine | 2.42 " |
| Sulphuric acid | 2.34 " |
| Silica | 14.65 " |
| PHOSPHORIC ACID | 35.43 " |

Sunflower oil is clear, pale yellow, almost inodorous, and of an agreeable mild taste. It is more limpid than poppy-seed oil. Its specific gravity is 0.9260 at 59° F. It solidifies to a white-yellow mass at —9.3° F. Five volumes of oil require for solution two volumes of ether. Alcohol absorbs only 0.62 per cent. It is miscible in all proportions with chloroform, benzol, etc.

The oil belongs to the medium drying oils. It contains, besides the glycerides of the acids related to linoleic acid, oleic and palmitic acids and only very little arachidic acid.

*Nitric acid* does not color sunflower oil; *nitric acid* containing some *nitrous acid* colors is slightly brownish-yellow, and *fuming nitric acid* reddish-brown, the oil becoming thereby only thick.

*Sulphuric acid* of 1.70 specific gravity colors the oil bluish-green, which changes to dirty gray. Stronger acid produces first a gray-brownish, and then a dark brown color.

A mixture of equal volumes of *sulphuric and nitric acids* causes only a dirty brownish color.

*Potash lye* and *ammonia* give white liniments.

| | |
|---|---|
| Saponification number of the oil | 193–193.3 |
| Iodine number of the oil | 128.4–129.9 |
| Iodine number of the fatty acids | 132.2–134 |
| Fusing point of the fatty acids | 73.4° F. |
| Congealing point of the fatty acids | 62.6° F. |

Adulterations of sunflower-seed oil are thus far unknown.

Cold-drawn sunflower oil is exclusively used as a table oil, and in Eastern Russia in the preparation of almost every kind of food. The warm-pressed oil is employed in the manufacture of varnish and of soap.

The sunflower is largely cultivated in the Government Saratow, Russia, and the seed expressed in the capital Saratow and its neighborhood. The stalks are used as fuel.

The oil is brought into commerce from Russia and Hungary.

2. *Madia oil* (*oleum madiæ; Madiaöl*, G.; *huile de madia*, F.) from *Madia sativa*, Molin., a plant indigenous to Chili. The seed contains from 35 to 40 per cent. of oil.

| | Madia seed. | Madia oil cake. |
|---|---|---|
| Oil | 38.82 p. c. | 9.46 p. c. |
| Organic substances | 49.58 " | 72.90 " |
| protein substances therein. | 19.80 p. c. | 31.00 p. c. |
| Ash | 4.20 " | 6.70 " |
| Water | 7.40 " | 10.94 " |
| | 100.00 " | 100.00 " |

The ash of the seed contains:

| | | |
|---|---|---|
| Potash | 12.66 | per cent. |
| Soda | 6.09 | " |
| Magnesia | 13.38 | " |
| Lime | 16.34 | " |
| Ferric oxide | 1.12 | " |
| Chlorine | 1.38 | " |
| Sulphuric acid | 1.62 | " |
| Silica | 2.13 | " |
| PHOSPHORIC ACID | 45.28 | " |
| | 100.00 | " |

Madia oil is dark yellow, has a peculiar but not disagreeable odor, and a mild taste. It does not keep well. The specific gravity of the crude oil is 0.9350 at 59° F. By refining with sulphuric acid the oil, which is quite thickly fluid, becomes more limpid, and has then a specific gravity of 0.9286 at 59° F. The cold-drawn oil solidifies at 14° F. and the warm-pressed at 5° to —4° F.

The oil is soluble in 30 parts of cold and in 6 parts of boiling alcohol and in equal parts of ether.

Its elementary composition is:—

| | | |
|---|---|---|
| Carbon | 76.00 | per cent. |
| Hydrogen | 12.15 | " |
| Oxygen | 11.85 | " |
| | 100.00 | " |

and it contains, besides palmitic, stearic, and oleic acids, an acid resembling oleic acid, which, however, possesses some drying properties.

The oil is intermediate between drying and non-drying oils. Exposed in thin layers to the air it requires months to become viscous, and while it absorbs considerable quantities of oxygen, it is thereby made only more thickly-fluid. Digested with plumbic oxide for some time it becomes almost colorless and thickens to the consistency of turpentine.

*Nitric acid* changes the oil but little; *fuming nitric acid* colors it brown-red.

*Sulphuric acid* of 1.70 specific gravity produces first a greenish color, which changes to dirty brown.

Shaken with *potash lye* and *ammonia* the oil gives yellowish liniments.

Saponified with *soda lye* it yields an odorless soap.

Madia oil is used principally as a lamp oil and lubricant, and in the manufacture of soap, though the cold-drawn oil is sometimes employed as a table oil.

3. *Niger oil, guizot oil or ramtil oil* (*oleum guizotiæ; Nigeröl,* G.; *huile de ram-till,* F.), from the seed of *Guizotia oleifera,* Cassiroi = *Verbesina sativa,* Roxb. = *Ramtilla oleifera,* D. C.; *Polymnia abessinica,* L., a plant indigenous to Abyssinia, and cultivated in India, and also in Germany and the West Indies. The seed is known in the London market as "*niger seed*" when brought from Africa, and as "*keersanee seed*" when imported from Bengal; it contains 40 to 45 per cent. of oil, and is pressed in England and in Germany. The yield obtained is 20 to 25 per cent. from the first cold pressure, and 12 to 15 per cent. from the second warm pressure.

| | Niger seed. | | Niger oil cake. | |
|---|---|---|---|---|
| Oil | 42.89 p. c. | | 5.36 p. c. | |
| Organic substances, | 47.08 " | | 76.50 " | |
| protein substances therein. | | 19.45 p. c. | | 32.60 p. c. |
| Ash | 3.61 " | | 8.30 " | |
| Water | 6.42 " | | 9.84 " | |
| | 100.00 " | | 100.00 " | |

The ash of the seed contains:

| | | |
|---|---|---|
| Potash | 18.64 | per cent. |
| Soda | 11.28 | " |
| Magnesia | 14.32 | " |
| Lime | 15.50 | " |
| Ferric oxide | 0.62 | " |
| Chlorine | 4.23 | " |
| Sulphuric acid | 4.00 | " |
| Silica | 8.16 | " |
| PHOSPHORIC ACID | 23.25 | " |
| | 100.00 | " |

The oil is yellow and has a peculiar nutty taste and odor, which are, however, not disagreeable. It is of the consistency of sesamé oil and has a specific gravity of 0.9242 at 59° F. It thickens at 17.5° F., forms a transparent yellowish mass at 14° F., and a solid white mass at 5° F.

Five volumes of oil require for solution 2 volumes of ether, and 100 volumes of alcohol dissolve 1 volume of oil.

The oil consists of the glycerides of oleic, palmitic, and myristic acids, and an acid belonging to the linoleic acid group. Its elementary composition is:—

| | |
|---|---|
| Carbon | 76.42 per cent. |
| Hydrogen | 11.82 " |
| Oxygen | 11.76 " |

It has but slight drying properties. Exposed in thin layers to the air it dries to a viscous, sticky mass, and when boiled with lead yields a poor quality of varnish.

*Nitric acid* colors the oil egg-yellow, and *fuming nitric acid* first reddish-yellow and later on red-brown. The oil does not solidify with nitric acid.

*Sulphuric acid* of 1.70 specific gravity gives a gray-green color; stronger acid first a greenish and later on a brown color.

A mixture of equal volumes of *sulphuric and nitric acids* colors the oil first dirty brown-yellow, which gradually darkens until, after a quarter to half an hour, the oil assumes a dirty black-brown color, which only changes back to red-brown after many hours. This is a characteristic phenomenon for niger oil.

*Zinc chloride* causes a very slight green color.

*Solution of nitrate of silver* is but very little reduced.

*Potash lye* and *ammonia* give yellowish liniments.

Saponified with *soda* the oil yields a beautiful hard soap.

Niger oil is used as a lamp oil and lubricant, and in the manufacture of soap. The poorer classes of India use it as a table oil.

The chief markets for niger oil are London, Hull, Hamburg, Magdeburg.

4. *Safflower oil (oleum carthami; Safloröl*, G.; ***huile de carthame***, F.) from the seed of *Carthamus tinctorius*, Linn., the safflower, the flowers of which are used as a dyestuff. The plant is cultivated in India, Egypt, and in Europe, especially in Germany. The seed contains 30 to 35 per cent. of oil, and is pressed or extracted especially in Egypt, the East Indies, and China. The oil is used as table and lamp oil, and medicinally against paralysis. It is quite limpid, pale yellow, and clear.

5. *Thistle oil* (*Distelöl*, G.; ***huile de chardon***, F.) from *Onorpodon acanthium*, Linn., the cotton thistle. The seed contains 30 to 35 per cent. of oil, with an obtainable yield of about 25 per cent. It is used as lamp oil.

6. *Burr oil* (*oleum bardanæ; Klettenöl*, G.; ***huile de bardanne***, F.) from *Arctium lappa*, Linn., the burdock. The seed contains 25 to 30 per cent. of oil, which is used as lamp oil.

### *Urticeæ* (*Urticinæ*).

*Hemp-seed oil* (*oleum cannabis; Hanföl*, G.; ***huile de chanvre***, F.) from the seed of *cannabis sativa*, Linn., the common hemp, indigenous to Central Asia and the East Indies, and cultivated in many parts of Europe and America. The plant which grows in India, and has been described by some botanists under the name of *Cannabis indica* or *Indian hemp*, does not appear to possess any specific differences from the common hemp, except that it is richer in resin. Most botanists have accordingly considered it identical with the *Cannabis sativa*, Linn. The resinous exudation removed from the leaves, slender stems, and flowers is called "*churus*," and forms a constituent of "hashish."

The seed contains from 30 to 35 per cent. of oil, of which about 25 per cent. are obtained by pressure and 30 to 32 per cent. by extraction.

| | German hemp seed. | | Russian hemp seed. | |
|---|---|---|---|---|
| Oil | 33.60 p. c. | | 31.42 p. c. | |
| Organic substances | 54.30 " | | 54.95 " | |
| albuminous substances therein | | 15.95 p. c. | | 15.00 p. c. |
| Ash | 3.45 " | | 4.50 " | |
| Water | 8.65 " | | 9.13 " | |
| | 100.00 " | | 100.00 " | |

### *Hemp-seed cake.*

| | German. | | Russian. | |
|---|---|---|---|---|
| Oil | 6.70 p. c. | | 7.45 p. c. | |
| Organic substances | 73.50 " | | 73.84 " | |
| albuminous substances therein | | 30.40 p. c. | | 30.50 p. c. |
| Ash | 8.20 " | | 7.86 " | |
| Water | 11.60 " | | 10.85 " | |
| | 100.00 " | | 100.00 " | |

On account of the hard hull enveloping the seed, the cake is of but little value as a feed.

The ash of Russian hemp-seed cake contains:

| | | |
|---|---|---|
| Potash | 19.13 | per cent. |
| Soda | 0.95 | " |
| Magnesia | 10.12 | " |
| Lime | 19.00 | " |
| Ferric oxide | 2.24 | " |
| Chlorine | 0.66 | " |
| Sulphuric acid | 2.06 | " |
| Silica | 8.53 | " |
| PHOSPHORIC ACID | 37.31 | " |

Hemp-seed oil freshly expressed has a mild odor, mawkish, unpleasant taste, and a greenish-yellow color turning brownish-yellow with age. It is 9.6 times more thickly fluid than water at 59° F. It thickens at 3° F., and forms a solid brownish-yellow mass at — 16.5° F. Its specific gravity is 0.9276 at 59° F. It dissolves in 30 parts of cold and in any quantity of boiling alcohol. A solution in 12 parts of boiling alcohol separates, on cooling, stearin. Two volumes of ether are required for the solution of one volume of oil. The oil extracted with canadol or carbon disulphide is brownish-yellow, and that extracted with ether intensely green when fresh.

The liquid fatty acids of hempseed oil consist essentially of linoleic acid and small quantities of linolic, isolinolic and oleic acids. The oil has an elementary composition of:

| | | |
|---|---|---|
| Carbon | 76.05 | per cent. |
| Hydrogen | 11.35 | " |
| Oxygen | 12.60 | " |
| | 100.00 | " |

*Nitric acid* colors the oil somewhat greenish-yellow, *nitric acid* containing some *nitrous acid*, reddish-yellow, and fuming nitric acid, dark brown-red.

*Sulphuric acid* of 1.70 specific gravity colors it greenish. Acid of 1.80 specific gravity gives first a more vivid green and later dirty brownish.

A mixture of equal parts of *nitric and sulphnric acids* colors the oil first greenish, next brownish, and finally black. The latter color holds for twenty-four hours, after which it changes to red-brown.

The temperature of a mixture of 15 grammes of hemp-seed oil and 5 grammes of sulphuric acid rises from 59° F. to 208.5° F.

*Concentrated hydrochloric acid* colors comparatively fresh oil grass-green, and old oil yellow-green; in both cases the color is characteristic.

*Zinc chloride* causes a yellow-green color.

*Solution of nitrate of silver* is not reduced.

*Ammonia* gives a greenish-whitish emulsion, and potash lye a greenish-yellowish one.

An adulteration of hemp-seed oil is readily recognized by the mixture of nitric and sulphuric acids, which, in the presence of foreign oils, does not produce a black color.

| | |
|---|---|
| Saponification number of the oil | 192–194 |
| Iodine number of the oil | 143–144 |
| Fusing point of the fatty acids | 66.2° F. |
| Solidifying point of the fatty acids | 59° F. |

Although hemp-seed oil possesses drying properties in a less degree than linseed oil, it is nevertheless used in the preparation of varnishes, especially the dark kinds.

As a lamp oil it has the same property as olive oil, of burning away very rapidly, and hence is chiefly used in the manufacture of soft soaps.

The principal market for hemp-seed oil is St. Petersburg, though occasionally it is also brought into commerce from German mills.

### *Juglandeæ* (*Amentaceæ*).

1. *Walnut oil, or commonly called nut oil* (*oleum juglandis; Wallnussöl*, G.; *huile de noix*, F.), from the kernels of the nuts of *Juglans regia*, Linn., or common walnut tree, indigenous to Persia, and cultivated in Europe and America. The nuts used for oil should be at least two or three months old, since the fresh kernels contain a kind of emulsion (milk), and give a turbid oil difficult to clarify. The kernels, which contain 40 to 50 per cent. of oil, are separated from the shells and skins, crushed and pressed. After the cold-drawn or "*virgin*" oil is obtained, the residue is pressed with the assistance of heat. The yield from the first pressure amounts to from 30 to 35 per cent., which is used as table oil, and from the second from 10 to 15 per cent.

| | Nut kernels. | Nut cake. |
|---|---|---|
| Oil | 48.65 p. c. | 5.25 p. c. |
| Organic substances | 38.20 " | 76.35 " |
| albuminous substances therein | 14.10 p. c. | 33.65 p. c. |
| Ash. | 2.30 " | 6.30 " |
| Water | 10.85 " | 12.10 " |
| | 100.00 " | 100.00 " |

The ash of the nut contains:

| | | |
|---|---|---|
| Potash | 31.10 | per cent. |
| Soda | 2.75 | " |
| Magnesia | 12.18 | " |
| Lime | 9.16 | " |
| Ferric oxide | 1.50 | " |
| Chlorine | 0.13 | " |
| Sulphuric acid | 0.18 | " |
| Silica | 0.52 | " |
| PHOSPHORIC ACID | 42.48 | " |
| | 100.00 | " |

Fresh cold-drawn nut oil is limpid, almost colorless or pale greenish-yellow, and has an agreeable taste and odor. It soon gets rancid, and in that condition possesses purging properties. Warm-pressed oil has a darker color and a peculiar acrid odor and taste. The specific gravity of the cold-drawn oil is 0.9250 at 59° F., and that of the warm-pressed oil 0.9268 at the same temperature.

Nut oil is 9.7 times more thickly fluid than water at 59° F. It remains limpid at 5° F., commences to thicken at 1.5° to —0.5° F., and solidifies to a solid white mass at —16.5° to —18.5° F.

One hundred volumes of cold alcohol are incapable of dissolving 1 volume of oil, while 60 volumes of hot alcohol dissolve 100 volumes of oil. Cold ether dissolves an equal volume of oil.

Nut oil consists of glycerides of linoleic, oleic, myristic, and lauric acids, and has the following elementary composition:—

| | | |
|---|---|---|
| Carbon | 77.46 | per cent. |
| Hydrogen | 11.83 | " |
| Oxygen | 10.71 | " |
| | 100.00 | " |

*Nitric acid* colors nut oil yellow, *nitric acid* containing some *nitrous acid* red-yellow, and *fuming nitric acid* dark red.

*Sulphuric acid* of 1.70 specific gravity causes first a brownish and then a brown color.

A mixture of equal parts of *sulphuric and nitric acids* produces first a brown-yellow and then a brown color.

*Zinc chloride* produces no effect whatever.

*Ammonia* and *potash lye* give yellowish-white liniments.

With *soda lye* the oil saponifies like linseed oil.

The principal adulteration of nut oil is with bleached linseed oil, which is readily recognized by the viscous resinous mass which is formed (see under *linseed oil*).

| | |
|---|---|
| Saponification number of the oil | 196–197 |
| Iodine number of the oil | 142–143 |
| Fusing point of the fatty acids | 68° F. |
| Solidifying point of the fatty acids | 60.8° F. |

With increasing oxidation of the oil the absorption of iodine decreases very rapidly.

When nut-oil is used as an adulterant of olive oil, the high absorption of iodine is compensated by an addition of lard oil olein.

Nut oil dries even better than linseed oil, and for that reason is preferred for fine oil painting. It gives varnishes which, on account of being entirely colorless, are in much demand for delicate white colors. It is occasionally used in the manufacture of soap and as lamp oil. It gives a beautiful, brilliant light.

Nut oil is brought into commerce from France and Switzerland in casks holding 110 lbs.

2. *Juglans nigra*, Linn., or black walnut, and

3. *Juglans cinerea*, Linn., or butternut. Both species are indigenous to North America. The nuts, though occasionally used for expressing oil, are of but little value for the purpose.

4. *Hickory oil* (*Hickoryöl*, G., *huile de hickory*, F.,) from the kernels of the nuts of *Carya alba*, Michaux; *Carya illionensis*, Nuttall; and *Carya olivæformis*, Nuttall, three species of hickory indigenous to North America. The oil resembles walnut oil, and is used as table and lamp oil. It is now brought into commerce under the name of American nut oil.

### *Abietineæ* (*Coniferæ*).

1. *Scotch fir-seed oil* (*oleum pini pingue; Kiefersamenöl*, G.; *huile de pin*, F.) from *Pinus sylvestris*, Linn. or the Scotch fir. The seed contains 25 to 30 per cent. of oil. The latter is brownish-yellow, somewhat thickly-fluid, and has an aromatic turpentine-like odor and taste. Its specific gravity is 0.9312 at 59° F., and at this temperature it is 11.4 times more thickly-fluid than water. It thickens at 3° F., assumes a whitish turbidity at —17.5° F., and solidifies at —22° F.

Pine-seed oil dries very quickly and furnishes an excellent varnish. It is also used as lamp oil.

2. *Spruce fir-seed oil* or *pitch-tree oil* (*oleum abietis seminis; Tannensamenöl*, G.; *huile de sapin*, F.) from *Abies pectinata*, D. C. = *Pinus picea*, Linn. = *Pinus abies*, Duroi, the spruce fir. The seed contains 25 to 30 per cent. of oil. The latter has an aromatic turpentine-like taste and odor, is clear and brownish-yellow, and has a specific gravity of 0.9250 at 59° F. It remains fluid at 5° F. and solidifies at —16.5° F. It does not dry as quickly as the preceding oil, as it contains a small quantity of resin in solution. It is used for the same purposes.

3. *Red pine-seed oil* or *pinaster-seed oil* (*oleum piceæ seminis; Fichtensamenöl*, G.; *huile de pinastre*, F.) from *Picea vulgaris*, Lamk. = *Pinus abies*, Linn. = *Pinus picea*, Duroi, the red pine. The seed contains 25 to 30 per cent. of oil. The latter is golden-yellow, has a taste and odor similar to that of the preceding and a specific gravity of 0.9288 at 59° F., being at this temperature 9.4 times more thickly-fluid than water. It thickens at 5° F. and solidifies to a white mass at —16.5° F. It is chiefly used for oil colors and occasionally as lamp oil.

4. *Cembra-kernel oil* (*Zirbelnussöl*) from *Pinus cembra*, L., the fir-leaved pine-tree or Siberian stone-pine, indigenous to the Eastern Alps and the Carpathians. The seed called *cembra nut* is edible, and the kernel, which amounts to more than one-half of the seed, contains 56 per cent. of oil consisting chiefly of oleic acid, very like linolein, and small quantities of myristicin.

## 3. *Glycerides of Physetoleic Acid = Train Oils.*

## OILS OF THE ANIMAL KINGDOM ONLY.

### A. SEAL OILS.

### *Pinnipedia* (*Mammalia*).

The *pinnipedia* or *pinnipeds*, embracing the seals and walruses, are commonly recognized by recent systematic writers as constituting a sub-order of the order *Feræ* or *Carnivorous Mammals*. Their whole form is adapted to life in the water, which element is their true home. Here they display extreme activity, but on land their movements are confined and labored.

The principal hunting-ground for seals is in the Behring Sea, in the waters between Greenland and the mainland of America and on the coast of Newfoundland. The yearly catch in all seas is estimated at one and a half millions of animals. On account of the indiscriminate destruction and consequent diminution, seal hunting is not so profitable as formerly. The simplest way of making seal oil is as follows: The fat adhering to the skin of the seal is detached from it, cut into pieces, and melted in caldrons, after which the oil is poured into barrels. This is the method often employed by the hunters. But the oil is also manufactured by steam in large establishments especially built for the purpose. They are particularly busy in spring, when whole cargoes of seal fat arrive, which is either boiled immediately, in order to extract the oil, or is stored away in long, floored cellars furnished with ventilators and windows. Large oak-wood tubs, lined with lead, and capable of holding 25,000 pounds of oil each, are placed at intervals in holes dug in the ground. The oil which runs out from the seal fat piled up in layers flows into these tubs by way of an inclined plane. The oil is then poured into barrels. In order that the skins from which the fat has not yet been removed may not spoil, they are salted again, just as it had been done on board the vessels, 5500 pounds of salt being generally used for salting 1000 skins, and only 2500

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pounds per 1000 for the final salting before the skins are stored in the cellars. The workman employed to detach the fat spreads the skin, with the fur down, on an inclined plank, which he lean against their breast, in order to have the free use of both hands. Then, armed with a two-handled knife, he scrapes the fat from the skin. The oil, which is pure and clear, running down during this operation, flows into a reservoir let into the ground. This work is extremely fatiguing. A strong and experienced workman can, however, clean 500 to 700 skins in a day.

The fat is then melted in large tubs, where it is exposed to the action of steam. The oil flows through a funnel-shaped apparatus and, finally, through pipes into immense oak-wood reservoirs. There are three such reservoirs connected by pipes and let into the ground, so that the oil from the first flows into the second and then into the third, from whence, through cocks, it passes into casks which can be shipped as soon as filled. The oil thus extracted forms the first quality. The second quality is obtained by melting the residue in caldrons and by pressing it. The color of this oil is dark brown. Before the residue is put in the caldrons it is thrown into a receptacle with an inclined bottom, and the whole mass is stirred violently by means of wooden shovels. This is done in the sunlight, so that the heat may help to melt the mass. This receptacle is joined to the caldron by a large gutter, which is walled up in the furnace. Through this gutter the residue is let into the caldron, there to melt, which done, the mass is taken out with dippers, cast into a box, and pressed. By means of the last operation all the remaining oil contained in the residue is extracted.

The residue and skeleton yield an excellent manure.

The pinnipeds constitute two minor groups or families:—

1. Walruses or *Trichechoidea*, of which there are two species, that of the Atlantic and that of the Pacific.

2. Seals or *Phocidæ*, of which there are about twenty-eight species.

*Walrus oil* (*oleum trichechi; Walrossthran*, G., *huile de morse*, F.) from *Trichecus rosmarus*, Linn., or the walrus. The length of a full-grown male animal is given at twelve to thirteen feet, and its weight at 2000 to 2500 pounds. The oil, of which a light and dark variety are brought into commerce, is valuable, but one animal seldom yields more than twenty to thirty gallons. It is less thickly fluid than seal oil, has a specific gravity of 0.9250 at 59° F., and separates stearin at 28.4° F. One hundred volumes of cold alcohol dissolves ten volumes of walrus oil, and the same quantity of hot alcohol ten to twelve volumes.

2. *Seal oil* (*oleum phocæ; Robbenthran*, G.; *huile de phogue*, F.) from the various species of *Phocidæ*.

a. *Archangel seal oil*, from *Phoca vitulina*, Linn., or the common seal, and some other varieties inhabiting the Baltic, North Sea, and White Sea.

It is brought into market as:—

| | Specific gravity at 59° F. |
|---|---|
| Pale Archangel seal oil | 0.9165 |
| Brown Archangel seal oil | 0.9170 |
| Sea-calf oil | 0.9155 |

Separates stearin below 37.5° F. One hundred volumes of cold alcohol dissolve one-half volume of oil, and one hundred volumes of hot alcohol eight to ten volumes. Reaction slightly acid. Principal markets Archangel, Copenhagen, Hamburg.

b. *Greenland seal oil*, principally from *Phoca greenlandica*, Müll., or the harp seal. Four varieties are brought into commerce:

| | Specific gravity. at 59° F. |
|---|---|
| Bright pale Greenland seal oil | 0.919 |
| Bright brown | 0.921 |
| Brown Greenland seal oil | 0.924 |
| Boiled seal oil | 0.926 |

Greenland "*three crowns*" oil is a mixture of several varieties of train oil, chiefly seal oil and shark oil and small quantities of whale and walrus oil. The seal oil used in the preparation of this oil is the so-called " fod" (foot), *i. e.*, the sediment formed by boiling and clarifying seal oil. It forms the principal constituent as regards quantity. On account of its fluidity and low specific gravity, the shark oil imparts to the "*three crowns*" oil its special properties.

Swedish "*three crowns*" oil is a mixture of various seal oils with ordinary fish oils. Both varieties are found in commerce adulterated with fat oils and resin oils. Their specific gravity is 0.923 at 59° F.

Greenland seal oils separate stearin below 41° F. One hundred volumes of cold alcohol dissolve 1 volume of oil, and 100 volumes of hot alcohol 9 volumes of oil. Seven and a half volumes of ether are required for the solution of 10 volumes of oil.

Principal markets, Copenhagen and Hamburg.

c. *Newfoundland seal oil*, from *Phoca lagura*, Cuvier, which is exclusively caught on the coast of Newfoundland. Three varieties are brought into commerce:—

| | Specific gravity at 59° F. |
|---|---|
| White inodorous Newfoundland | 0.9270 |
| Yellow Newfoundland | 0.9270 |
| Brown Newfoundland | 0.9275 |

Solidification point below 39° F. Principal markets, Glasgow, Leith, and London.

d. *South Sea seal oil*, from *Phoca proboscidea*, Linn., or *Macrorhinus proboscideus*, Cuv., or the Southern sea elephant. Two varieties are known in commerce:—

| | Specific gravity at 59° F. |
|---|---|
| Pale South Sea seal oil | 0.921 |
| Brown South Sea seal oil | 0.930 |

The animals, which yield an astonishing quantity of almost inodorous oil (one animal as much as 2400 pounds), are chiefly taken by American vessels. New York and London are the principal markets for the oil.

e. *Caspian seal oil.* from *Calocephalus caspicus*, Cuv., a variety of seal inhabiting the Caspian and other large inland seas of Asia. The oil is exclusively used in Russia. Principal market, Astrachan.

All walrus and seal oils have a disagreeable odor; the dark brown varieties, which drain from the putrid mass after the pale oil has been obtained, are the most strongly scented and nauseous. They contain no albuminous substances like vegetable oils, but animal glue, which can be precipitated with tannin and metallic salts. They have a specific gravity of 0.915 to 0.930 at 59° F., and are but sparingly soluble in alcohol, while of ether they require almost an equal volume for solution. Some of the oils separate stearin below 41° F.. while others solidify to a solid mass only at 28.5° to 26.5° F.

While fresh the oils show only a slight acid reaction, the acidity, however, increasing with age. They contain principally glycerides of physetoleic acid, $\left.\begin{matrix} C_{16}H_{29}O \\ H \end{matrix}\right\} O$, of stearic and palmitic acids, a small quantity of oleic acid, and traces of butyric, valerianic, etc., acids.

Pale brown Greenland seal oil is composed of:—

| | | |
|---|---|---|
| Carbon | 77.10 | per cent. |
| Hydrogen | 13.50 | " |
| Oxygen | 9.40 | " |
| | 100.00 | " |

*Fuming nitric acid* produces a red-brown color in all seal oils.

*Sulphuric acid* of 1.65 to 1.70 specific gravity causes first a red-yellowish color, then a reddish-brown, and finally a brown-red.

*A mixture of equal volumes of nitric and sulphuric acids* produces first a reddish, and next a brown color, the transition to brown taking place very rapidly.

The adulteration of seal oil with other train oils and rosin oil, to which it is chiefly restricted, is recognized, on the one hand, by the greater or lesser solubility in alcohol, and on the other, by the incomplete saponification.

## B. WHALE OILS.

### *Cetacea (Mammalia Natantia).*

These oils are obtained from the *cetacea*, an order of vertebrated mammiferous marine animals. They are divided into two families:—

1. *Sirenia*, or sea cows.
2. *Cetacea*, or whales, which are subdivided into
   a. *Delphinodea*, or dolphins.
   b. *Balænodea*, or whales.

After the capture of the animals the blubber is removed and cut into pieces, which are either at once melted down in large boilers on board the vessel or packed into casks with wire-work bottoms for transportation to the sea-ports. During the voyage a decomposition of the animal substances takes place by the destruction of the cell tissues of the blubber, which, though it assists in the extraction of the oil by allowing a part of it to drain off, gives rise to a very unpleasant odor, due to the development of phocenin, a substance not yet thoroughly known. The residue of cellular blubber remaining in the casks is melted down in special establishments. The oil drained off during the voyage is purified by passing steam through it.

The manner of rendering the blubber on board the vessel is as follows: When the entire blubber is on board, the large pieces, called "blanket pieces," are cut into "horse pieces," which are about fifteen to eighteen inches long and six to eight inches in breadth and thickness. Any flesh, termed lean or fat lean, that may adhere to the horse pieces, is cut off, and being thus prepared, the blubber is transported in strap tubs to the mincing horse, where the ordinary two-handled knife is used, or else is taken to the mincing machine, where it is cut crosswise into slices a quarter of an inch in thickness; then it goes into a large mincing tub, which completes its preparation for boiling. From the mincing pots it is pitched into the try pots with the blubber fork, where the oil is extracted by boiling. During this process the minced blubber is frequently stirred to prevent it from burning and settling on the bottom of the pots, and when the scraps are sufficiently browned to show that the oil is well tried out, they are skimmed off with a skimmer into a receptacle called the scrap hopper, and after the oil drains off from them they furnish an abundant supply of fuel for the fires.

From the pots the oil is bailed into a large copper tank, called a cooler; from the cooler it passes through a cock into the deck pot, and from the deck pot the casks are filled. When a cask is full it is rolled off and headed up, and in rough weather it is lashed to the ship's rail for greater security. The oil, after passing through the cooler and deck pot, is still very hot if the blubber is being tried out rapidly, and for this reason the casks, when first filled, require the constant attention of the cooper, as the hoops have to be frequently driven to prevent leakage. The oil taken from the case of the sperm whale is sometimes put into casks without boiling, but usually it is carefully scalded with the junk and a portion of the hump, and when put into casks is known as "head" or "head matter." After the oil becomes cool it is run through a hose into empty casks, which have been prepared with great care and securely stowed in the hold, or they are lowered and stowed after being filled on deck.

### a. *Sirenia or Sea Cows.*

*Dugong oil*, from *Halicor Indicus*, Desm., or the dugong. This animal, which is the eastern representative of the family of *Sirenia*, is distributed over the Indian Ocean and is found principally amongst the islands of the Archipelago and the northeastern coast of Australia. In length it varies from six to fifteen feet. The weight of an ordinary specimen will be from four to six hundredweight, although individuals weighing something like a ton and producing no less than twenty gallons of purified oil have been caught. The dugong is taken either with nets or by harpooning. When one has been caught it is immediately brought ashore and cut up. The oil is then extracted and subjected to a variety of complicated processes, finally undergoing a chemical clarification which renders it fit for the European and American markets, where, owing to its medicinal properties, there is a large demand for it.

Two representatives of the *Sirenians* are found upon the American coast, *Trichechus manatus*, Linn., or South American manatee, and *Trichechus latirostris*, True, or Florida manatee. They are used for food and hunted for their fat, each animal yielding from five to twenty gallons of excellent oil.

### b. *Delphinodea or Dolphins.*

1. *Porpoise oil* (*oleum phocaenae; Meerschweinthran*, G.; *huile de marsium*, F.) from *Delphinus phocaena*, Linn. = *Phocaena communis*, Cuv., or the porpoise. This animal is, as a rule, from 4½ to 6 feet long, seldom 7½ feet. It lives in schools in all European seas, though its actual home is the Northern coast of the Atlantic Ocean. When caught the entrails are removed, the body cut up and boiled for obtaining the oil. The oil is pale yellow to brown and has a fishy odor, which it, however, loses on exposure to the air. When fresh it is indifferent to litmus paper, but absorbs acid properties from the air. Its specific gravity is 0.9220 at 59° F. It solidifies at 3.2° F. Cold alcohol dissolves 1.20 per cent. and boiling alcohol 20.0 per cent. of oil. The oil consists of glycerides of physetoleic, oleic, stearic, palmitic, and valerianic acids.

2. *Black fish oil* (*oleum delphini; Delphinthran*, G., *huile de dauphin globicephale*, F.) The oil is pale yellow, and has a strong, fishy odor. Neither the fresh oil nor an alcoholic solution of it shows an acid reaction. The specific gravity of the oil is 0.9180 at 59° F. One hundred parts of boiling alcohol of 0.812 specific gravity dissolve 40 parts of blackfish oil, the solution becoming turbid at 122° F. One hundred parts of boiling alcohol of 0.975 specific gravity dissolve 60 parts of the oil. Exposed to a low temperature blackfish oil separates spermaceti at from 41° to 26.5° F. The oil freed from spermaceti is still more soluble in boiling alcohol, 100 parts dissolving 100 parts of oil. The solution shows a slight acid reaction, which, however, disappears by adding water.

The oil brought into market under the name of porpoise-jaw oil, blackfish melon oil, watch oil, and clock oil, are obtained from the blackfish. Porpoise-jaw oil, or, more correctly, blackfish-jaw oil, is taken from the blackfish by a method known to a few persons only. It can be refined a little by exposure to the cold at zero, and in that state, with the atmosphere at zero, it is strained through a cotton flannel strainer made in the shape of a cone, but when filtered through paper it is so limpid that it has no lubricating properties whatever and becomes useless. It is warranted not to corrode on brass or rust on steel, and will not glue on the finest watch. It will not congeal at zero, though it will thicken and turn a little milky in appearance.

Blackfish melon oil derives its name from the mass taken from the top of the head of the animal, reaching from the spout-hole to the end of the nose and from the top of the head down to the upper jaw, from which it is extracted. When taken off in one piece this mass represents a half watermelon, weighing about twenty-five pounds. When the knife is put into the centre of this melon the oil runs more freely than the water does from a very nice watermelon. It is claimed to be superior to jaw oil in every repect. Since the year 1842, Caleb Cook, of Provincetown, Mass., claims to be the only person who understands the art of producing this oil free from all glutinous matter and fit for use. This, he says,* is done by a process known only to himself; not by mixing other oils and liquids with it, but by extracting all the acid and gluten from it and leaving the oil pure for the finest and most delicate machinery. This, he says, cannot be done by the chilling and straining process, for when it becomes perfectly transparent at zero the lubricating properties are all gone, the oil runs off the pivots, spreads upon the plates, dries up, the pivots cut, turn red, and the oil is worse than worthless.

Porpoise-jaw oil and blackfish melon oil are worth from five dollars to fifteen dollars per gallon according to supply. They are largely used by manufacturers of fire-arms, watches, and philosophical apparatus, and in government light-houses for the clocks of revolving lights. The color of the oils is very light, and can be made very white by placing in the window, where they will bleach in a short time. One drop of water in one pint of oil will injure it very much.

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*Proceedings United States National Museum, Vol. 1, 1879.

3. *Delphinus delphis*, Linn., the common dolphin.

5. *Delphinapterus leucas*, Pall., the white whale, white fish, or beluga, inhabits the seas above 50° north latitude. It is 16 to 19 feet long, measures 9 feet in circumference, and weighs 3300 to 3500 lbs. It yields from 400 to 500 lbs. of oil, which is esteemed of greater value than seal or walrus oil.

5. *Tursio vulgaris* = *Delphinus tursio*, Linn., is found in all seas of the northern hemisphere.

6. *Monodon monoceros*, Linn., the narwhal, is found in the northern seas and grows to 20 feet in length. The male has usually one long twisted tusk projecting from the upper jaw like a horn. It yields a very white oil, and its meat is highly esteemed by the inhabitants of Greenland. The tusk is worked like ivory.

7. *Sperm whale oil* or *cachalot oil* (*oleum ceti; Pottwalthran*, G.; *huile de cachalot*, F.) from *Physeter macrocephalus*, Linn., the sperm whale. This whale inhabits all seas up to 70° northern and southern latitudes. The male attains a length of from 64 to 78 feet and a girth of 39 feet. The length of the head amounts to ⅓ the length of the body. The female attains only half the size of the male. A large sperm whale may yield 70 to 90 tons of oil and 50 cwt. of spermaceti. The oil is one of the most valuable of commerce, the amount annually procured being limited. It is quite limpid, has a specific gravity of from 0.875 to 0.883, and commences to separate spermaceti at 43° F. and stearin at 17.5° F. Ten volumes of alcohol dissolve two volumes of oil. With ether it is miscible in all proportions. Of all the train oils it alone solidifies somewhat with the elaïdin test.

Alfred H. Allen, in "Commercial Organic Analysis," Vol. II., gives the following rules for the examination of sperm oil:

1. Note the specific gravity. If of less density than the above figures, the only possible adulterants of the sample are mineral oils, shark liver oil, and African fish oil. These admixtures may be detected by the following tests:—

2. Compare the viscosity of the sample with that of a specimen of genuine sperm oil.

3. Note the heat produced on admixture with sulphuric acid. African fish oil, which is a common adulterant of sperm oil, but a very bad lubricant, causes a rise of temperature through more than twice the number of degrees observed with pure sperm oil.

4. Note the color produced with sulphuric acid. Sperm oil is colored far less intensely than other marine animal oils, while shark liver oil gives a violet coloration.

5. Examine the spectrum of the oil. Vegetable oils may be readily detected by their well-defined absorption bands, which are absent from sperm and other fish and animal oils.

6. Observe the drying properties of the oil.

7. A very valuable means of detecting and determining other fat oils when present in sperm oil is based on the neutralizing power of the sample when examined by Koettstorfer's method. As 1000 parts of sperm oil neutralize only 130 to 134 parts of caustic potash (KHO), while other oils require from 176 to 197 parts of potash, the proportion of foreign fatty oil in sperm oil may be very approximately ascertained by the equation

$$F = (P - 132) + 1.851;$$

in which P is the nnmber of grammes of caustic potash required for 1000 of the sample, and F is the percentage of foreign fat.

8. Owing to the fact that hydrocarbon oils have no neutralizing power on alkali, it would be possible to concoct a mixture of mineral oil and rape or lard oil which should be undistinguishable from sperm oil, either by the above test (No. 7) or by that of specific gravity. If any such complex adulteration be suspected, it is desirable to employ the following method: Saponify 5 grammes of the sample, agitate the solution of the soap with ether, separate and evaporate off the ether,

and weigh the residual unsaponified matter. Pure sperm oil, when thus treated, yields from 39 to 41 per cent. of unsaponifiable matter of a pale yellow color,* while no other animal or vegetable oil is known to yield more than 1½ or at most 2 per cent. to ether. Hence, if the adulterant be a fatty oil only, the proportion present is readily ascertained from the diminished quantity of this matter obtained. This curious substance, which was first discovered by the author, may be termed "spermyl alcohol," and appears to replace the glycerin obtained by the saponification of ordinary oils. In short, sperm oil appears to be a fluid wax, rather than a true oil. Sperm oil yielding no glycerin, the sum of the spermyl alcohol and fatty acids obtained should amount to more than 100.00 in a pure sample, and hence the proportion of foreign fat oil and mineral oil in sperm oil can be deduced from a consideration of the analytical data. Thus:

| Oils. | Products of the saponification of 100 parts of oil. | | |
|---|---|---|---|
| | Fatty acids. | Ether residue. | Glycerin. |
| Pure sperm oil ................ | 60 to 64 | 39.5 to 41.5 | None. |
| Other fat oils ................ | 95 to 96 | .5 to 1.5 | 10.4 |
| Mineral oil ................ | None. | 100 | None. |
| Rape oil, 60<br>Mineral oil, 40 } ................ | 57.6 | 40 | 6 |

From these figures it will be seen that an admixture of foreign fat oil would increase the percentage of fatty acids in almost the same proportion as the adulterant used, the ether residue being correspondingly reduced. By a judicious mixture of rape and mineral oils the analytical results of sperm oil might be approximately simulated, but glycerin would be produced, and the sum of the fatty acids and ether residue would be sensibly *less* than 100. Besides, to obtain a mixture of the same density as sperm oil, so very light a mineral oil would require to be used that it would necessarily be liquid, even at 0° C. (32° F.), and would have so low a flashing point that it could without difficulty be detected in, and even distilled out of, the original oil or the ether residue. Sperm oil does not flash below 220° C. (428° F.).

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* This substance is solid at the ordinary temperature, but melts very readily, and distils apparently unchanged at a very high temperature.

The head cavity of the sperm whale, called by the whalers the case, contains an oily fluid which, after death, concretes into a granulated substance of a yellowish color, called *spermaceti* (see waxes).

The spermaceti when subjected to pressure yields a fluid portion which is called *spermaceti oil* (*oleum spermaceti; Walratöl*, G.; *huile de spermaceti*, F.). It has a yellow color, a somewhat disagreeable, peculiar odor, a specific gravity of 0.910 at 59° F., remains liquid at —0.4° F., saponifies with difficulty, and appears to be isomeric with spermaceti.

Besides sperm oil, spermaceti, and spermaceti oil, the sperm whale furnishes ambergris, a highly valued substance used in the manufacture of perfumery. It is a fatty, waxy substance found in the cæcum, and, as supposed by some, as a morbid secretion of the urinary bladder. According to Mr. Beale, it consists of the mere indurated fæces of the animal, perhaps somewhat altered by disease. It is often found floating on the sea on the coasts of Arabia, Madagascar, Japan, etc. It has a gray-white color, often with a black streak; has a strong but rather agreeable smell, a fatty taste, is lighter than water, melts at 140° F., dissolves readily in absolute alcohol, in ether, in both fat and volatile oils. It contains 85 per cent. of the fragrant substance called ambreine. This is extracted from ambergris by digestion with alcohol, filtering the solution, and leaving it to spontaneous evaporation. It is thus obtained in the form of delicate white tufts, which are converted into ambreic acid by the action of nitric acid. Sperm oil is chiefly found in English and American commerce.

### B. *Baleaenodea = Whales, Whalebone Whales.*

1. *Doegling oil (oleum chenodelphini; Doeghlingthran,* G.; *huile de rorqual rostré,* F.) also called *Arctic sperm oil* and *true bottlenose oil,* from *Chenodelphinus rostratus,* Br., and from *Balaenoptera physalus,* Gray= *Balaenoptera rostrata,* Fab., = *Balaena rostrata,* Ch., the first a connecting link between the dolphins and whales, and the latter the smallest of the finback whales. Doegling oil is colorless to brown and of a slightly nauseous odor. It has a specific gravity of 0.905 at 59° F., is limpid and absorbs oxygen, becoming thereby more thickly fluid and of a greater specific gravity. It remains limpid below 41° F,, becoming only slightly turbid. Below 28.5° F. it solidifies to a paste. It dissolves in 25 parts of cold and in 2½ parts of boiling alcohol.

Treated with *nitric acid* it shows an inclination towards solidifying.

It contains only about half as much oxygen as the other train oils:

| | | |
|---|---|---|
| Carbon | 79.87 | per cent. |
| Hydrogen | 13.36 | " |
| Oxygen | 6.77 | " |
| | | " |
| | 100.00 | " |

and besides physetoleic acid, etc., spermaceti, of the latter about 1 per cent. The *doeglic acid* $\left.\begin{matrix}C_{19}H_{35}O\\H\end{matrix}\right\}O$ found by Scharling is a mixture of physotoleic acid with an allied acid, as shown by the formula, with odd carbon atoms.

Doegling oil is chiefly used as a lubricant and for hardening steel weapons. It possesses great power of penetrating the epidermis, the interesting experiments of Prof. Guldberg of Christiania, showing that in this respect it far surpasses olive oil. Moreover, the Northmen have known its properties for centuries. This the *Speculum regale,* an old manuscript written in Norway about 1250, in speaking of various kinds of train oils, mentions that the flesh of these whales is not fit to eat, because the fat rendered from them cannot be digested by men or animals; it penetrates everywhere through the body and also through wood, as well as through a horn, if kept in it for some time.

Lucas Jacobson Debes narrates, in 1673, that doegling fat penetrates through the pores of the skin and colors the clothing yellow. Olaffen and Povelsen also speak of the penetrating power of doegling oil.

Finally Prof. Guldberg, during his travels in Finland, 1881 to 1893, observed that the men engaged in cutting up dead, frequently half-putrid whales, when cutting themselves never suffered from local inflammation, and successfully made the test on himself. Dr. C. Boeck recommends the oil for chloroform mixtures, for mercurial ointment, and in combination with lead plaster, salicylic acid, $\beta$-naphthol, aristol and iodoform.

The principal market for doegling oil is Copenhagen.

2. *Humpback oil* or *finback oil* (*oleum balaenopteræ; Finnfishtkran*, G.; *huile de rorqual*, F.) from *Balaenoptera longimana*, Rud. = *Balaenoptera boops*, Fab. = *Rorqualus boops*, Cuv., the hump-backed whale. The oil comes into commerce from almost colorless to brown, and the specific gravity of the different kinds ranges from 0.915 to 0.920 at 59° F. The oil separates some stearin at 46.5° F., and becomes completely thick at 26.5° F. The dark varieties have a peculiar, very disagreeable odor. With ether the oil is miscible in all proportions.

The dark oil has the remarkable property of mixing to a clear solution with nearly equal volumes of alcohol, but the mixture soon separates into oil and an alcoholic solution of oil, so that ten volumes of alcohol dissolve four volumes of oil. By again mixing the same fluid, the mixture remains turbid and clarifies later on into the originally employed volumes of oil and alcohol, the latter containing some stearin and spermaceti.

The oil is composed of:—

| | | |
|---|---|---|
| Carbon | 77.05 | per cent. |
| Hydrogen | 12.05 | " |
| Oxygen | 10.90 | " |
| | 100.00 | " |

Most of the oil is brought into commerce from Norway, France taking the greater portion of it.

3. *Right whale oil*, or *train oil*, (*oleum balænæ; Walfischthan*, G.; *huile de baleine*, F.) from *Balæna Mysticetus*, Linn., the Greenland whale or the right whale. The oil, when well prepared, is of a honey-yellow color, and has a fishy odor and taste. It has a specific gravity of 0.925 to 0.927 at 59° F. It commences to separate some crystals at 50° F., which increase with a decreasing temperature, and at 28.5° F. nearly all the stearin and some spermaceti are separated. Two volumes of hot alcohol dissolve five volumes of oil. With ether the oil is miscible in all proportions. When heated for some time at nearly 382° F., the oil becomes black under decomposition. It is composed of:—

| | | |
|---|---|---|
| Carbon | 76.85 | per cent. |
| Oxygen | 11.80 | " |
| Nitrogen | 11.35 | " |
| | 100.00 | " |

Principal markets Hamburg, Copenhagen, New York.

All whale oils when fresh are neutral, but later on slightly acid. Like the seal oils, they show the same behavior towards reagents and can therefore be classed together.

*Fuming nitric acid* produces first a brownish coloring with a very slightly bluish hue; the color next changes to brown, and finally to black-brown.

*Sulphuric acid* of 1.65 to 1.70 specific gravity causes a brown color, which later on becomes black-brown.

A mixture of equal volumes of *nitric and sulphuric acids* with an equal volume of oil gives first a yellow color, then a reddish one, and finally a dirty brown one.

*Soda lye* and *syrupy phosphoric acid* color all train oils red-brown.

The various whale oils which separate stearin and some spermaceti near the freezing point are used in the manufacture of various commercial varieties, which are chiefly brought into commerce from Norway.

Thus the pure whale oils, pale, brownish-yellow or brown, are dealt in according to the manner of preparation as "*unpressed whale oils.*" If the oils are cooled below the freezing point of water for the separation of stearin, etc., the solid and expressed separations represent " whale fat " or " whale tallow." These separations are white or yellow, with a fusing point between 82° and 100° F. and are used in the manufacture of soap. The palest varieties of oil freed from stearin—the "*pressed whale oils*"—are frequently incorrectly called "spermaceti oils."

## C. LIVER OILS.

### *Gadini (Pisces).*

The codfish and its allies constitute, from an economical point of view, the most important of all families of fishes, containing, as this family does, a large number of species, most of them of considerable size, distributed throughout all parts of the northern hemisphere, usually found together in great numbers, readily captured and easily preserved for future use.

1. *Cod liver oil* (*oleum jecoris aselli*; *Leberthran*, G.; *huile de foie de morue*, F.) from the different varieties of *gadus*, or the codfish. The preparation of cod-liver oil is carried on on an extensive scale, especially in Norway, Newfoundland and Scotland.

The codfish, *gadus morrhua*, appers at Bergen, Norway, and farther north, in the months of January and February, and is caught in quantities of six to eight millions or more. A little later in the year, in March, the codfish appears at Lofoden, where sometimes upwards of twenty-four millions are caught. Later on the fish goes farther north to Finmark, where the catch is quite as large as at Lofoden. The number of fishermen who congregate at Lofoden is about sixteen thousand. As soon as possible the fishes are brought on shore and sold to the tradesmen, manufacturers, etc., or, what now happens very seldom, the oil is extracted by the fishermen themselves. There are five different kinds of cod-liver oil:—

1. That extracted by steam; the *white* oil.
2. The ordinary medicinal oil, of a *light yellow* color.
3. Oil of a little *darker yellow* color.
4. The *brown* medicinal oil.
5. The *dark brown* or tanner's and currier's oil.

*Steam oil.*—For this the livers are subjected to the action of steam heat in pans, pressed and transferred to closed lead cisterns, where the oil is left to deposit stearin and impurities. After one or two months the clarified oil is bottled. Of course, the higher the temperature at which it is bottled the more liable it is to deposit stearin at a lower temperature, and hence the oil merchants draw off the oil at as low a temperature as possible.

*Light yellow medicinal oil* is prepared by the old process; the livers are allowed to remain in heaps at ordinary temperatures until the oil runs out by itself. It is evident that through this long exposure to the air the oil gets somewhat rancid, and of course stronger in taste and smell than the steam oil. Here, as elsewhere, the chief difference is due to the more or less cleanliness observed. Livers kept in new oaken vats, the oil being drawn off frequently, will always yield milder oil than when treated in old, rancid casks, especially, if the product is kept in old tar or paraffine barrels. The truth is that the oil dealers do not pay a single cent more to the oil pressers for carefully-made oil than for the ordinary article, hence there is but little inducement for taking extra care and trouble. Being prepared without heat, this second-class oil contains very little stearin. The third and fourth qualities,

*Darker yellow and brown medicinal oils*, are prepared by pressing the livers and applying a little heat. Dr. de Jongh's oil is of the fourth kind. He treats it very carefully and bottles it perfectly clear, and, of course, charges a high price. The fifth quality,

*Currier's oil*, is obtained by letting the livers ferment and heat, and then submitting them to strong pressure.

Contrary to the generally received opinion, in Norway itself very little, if any, adulteration takes place. Very few other oils are to be had there to use as adulterants, and what might be employed for the purpose can only be procured in very small quantities, so that adulteration is not there profitable.

The only oil which is often sold for cod-liver oil is the oil of the liver of the *Scymnus borealis*, a fish belonging to the shark family, found in large numbers in the Arctic Ocean, the northern portion of the North Sea, and along the west coast of Norway. One single fish yields two hundred to three hundred pounds of oil. Of late this oil, which contains very little stearin, is very light in color, and is cheaper than cod-liver oil, has found ready sale. It is said to be very difficult of digestion.

In Scotland the fresh, thoroughly cleansed livers are cut in small pieces and heated, under constant stirring in iron boilers, to from 176° to 194° F. until converted into a pasty mass. This is ladled into muslin bags and pressed while hot. The pulpy mass left in the bags is subjected to stronger pressure. At 59° to 61° F. the oil separates considerable stearin, which is removed by filtering.

The far-famed cod-liver oil of Portugal Cove, Newfoundland, is prepared as follows: The livers are first carefully washed and must be "cooked" at once while fresh. They are first put into a large tin boiler. This is plunged into a larger iron boiler filled with hot water, the water not being allowed to touch the livers, which are thus gently steamed till a quantity of oil is floating on the surface. This is dipped out and filtered through blanketing first, then twice afterwards through bags of moleskin. From the last filtration it comes out of a beautiful crystalline transparency and without any unpleasant smell and taste. The oil is now poured into sixty-gallon casks and forwarded to the exporting merchant. On an average it requires two and a half gallons of livers to produce a gallon of oil. The refuse is placed under screw presses and the remainder of the oil extracted. This is not refined, but sold as common oil, and is used largely on railways and for lubricating machinery. The secret of making good cod-liver oil lies in the application of the proper degree of heat—too much or too little will seriously injure the quality. Great attention to cleanliness is also necessary, the filtering bags requiring to be washed thoroughly every day and the troughs scrubbed out with great care.

Light yellow cod-liver oil shows a slightly acid reaction and has a specific gravity of 0.923 at 59° F. In cold alcohol 2.5 to 2.7 per cent. of oil is soluble, and in hot alcohol 3.5 to 4.2 per cent.

Darker yellow and brown cod-liver oil reddens litmus paper more than the preceding variety, and has a specific gravity of 0.925 at 59° F. Cold alcohol dissolves 2.5 to 3 per cent. of the oil, and hot alcohol 5 to 6 per cent.

Currier's oil, which is principally used in tanneries, reddens litmus paper very strongly. Its specific gravity is 0.929 at 59° F. Cold alcohol dissolves 5.7 to 6.5 per cent. and boiling alcohol 6.5 to 7 per cent. In ether it is soluble in all proportions.

The elementary composition of cod-liver oil is:—

| | | |
|---|---|---|
| Carbon | 75.91 | per cent. |
| Hydrogen | 12.22 | " |
| Oxygen | 11.87 | " |
| | 100.00 | " |

It is a mixture of the most manifold glycerides, as that of oleic, physetoleic, palmitic, stearic, and myristic acids. It contains besides free acids, such as acetic and butyric acids; further, gallic acids and constituents of gall—fellic, cholic and bifellic acids, biliverdin, bilifuscin. Of bases it contains small quantities of trimethylamine, tertylamine, amylamine, hexylamine and a new base hydrotoluidine, boiling at 388° to 392° F., as well as two non-volatile bases—asseline and morrhuine.

The gaduin of De Jongh and the gadic acid of Luck are not independent bodies. The same may be said in reference to experiments communicated by Chapoteaut, as regards the separation of a body called "morrhuol" from cod liver oil. According to these experiments, the cod liver oil to free it from free acids is first treated at a low temperature with soda solution and then agitated with 90 per cent. alcohol, when the alcoholic fluid is lifted off and subjected to distillation. The residue from the distillation—the morrhuol—is claimed to contain the effective constituents of cod liver oil, while the oil extracted with alcohol is almost odorless and differs but little from other fats. The yield varies, brown oils giving 4.5 to 6 per cent. and morrhuol, yellow oils 2.5 to 3 per cent. and white oils 1.5 to 2 per cent. Morrhuol is said to be a liquid of a sharp, bitter aromatic taste, which partially crystallizes at the ordinary temperature. It is claimed to contain 10 to 12 times more combinations of phosphorus, bromine and iodine than cod-liver oil.

Cod liver oil is remarkable not only for a multiplicity of organic substances, but also for a number of inorganic combinations, for it contains small quantities of bromine, iodine, sulphur, and combinations of phosphorus, besides sulphates and phosphates of lime, magnesia, and soda, the attention of chemists having been especially attracted to the small quantity (at the utmost 0.01 per cent.) of iodine. It may be remarked that iodine is not contained in the oil in such a condition as to allow of its extraction by water or spirit of wine, or its detection by the ordinary reagents. The sample should be saponified by trituration with caustic soda or potash lye and hot water, the resulting soap cautiously dried and incinerated, the ashes digested with water, and filtered. The filtrate is then concentrated, and on addition of dilute sulphuric acid, starch paste, and chlorine water, the presence of iodine is indicated by the well-known blue coloration.

From 1873 to 1883, the catch of codfish amounted to 61 per cent. of the value of the total Norwegian catch of fish.

The first steam factory for the production of cod liver oil was established in 1850, by Peter Möller. The output amounted to

20 tons of cod liver oil, while the 200 to 300 establishments in existence, in 1887, produced 20,000 tons.

2. *Coal fish oil, merlan oil,* or *pollack oil* (*oleum jecoris merlangi; Sejthran,* G.; *huile de foie de merlan,* F.) from the livers of the *Merlangus* species. The pollack, *Merlangus carbonarius,* Cuv., or *Pollachius carbonarius,* is the coal fish of England and the *sei* of Norway and Sweden. It is much esteemed for food, and especially for the oil prepared from the liver. The liver yields a great quantity of oil, proportionally much more than that of the cod. It is probable that much of the cod liver oil in the market is more or less adulterated with pollack-liver oil. No one has yet demonstrated that its medicinal properties are inferior. It is prepared in the same manner as cod liver oil. The best variety is paler in color than cod liver oil and the brown variety darker. While cod liver oil remains limpid and clear at a moderate degree of cold, pollack liver-oil becomes stiff and granular. Its odor and taste are the same as those of cod liver oil. Its specific gravity is 0.927 at 59° F. Cold alcohol dissolves 3.4 per cent., and hot alcohol 6.5 per cent. Its chemical composition is the same as that of cod-liver oil.

## *Plagiostomi* (*Pisces*).

### a. *Squalini or Sharks.*

*Shark oil* (*oleum squali; Haifischthran,* G.; *huile de selacke,* F.) prepared from the livers of various species of the shark. One liver yields from fifteen to sixty gallons of oil. Shark oil is distinguished as being the lightest of the fixed oils, its specific gravity ranging from 0.870 to 0.875, or at the utmost to 0.880 at 59° F., so that a mixture with other train oils can at once be recognized by the higher specific gravity. The odor and taste of the oil are not very nauseous, but rather peculiar and very acrid. It is pale yellow and clear, and remains fluid at 21° F. Ten volumes of cold alcohol dissolve 1 volume of oil, and 1 volume of hot alcohol 4 volumes of oil; of ether the oil requires equal parts for solution. The oil burns with a bright flame without carbonizing the wick.

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Shark oil contains the same constituents as cod-liver oil, but is richer in iodine. Besides being employed in the adulteration of cod-liver oil, it is largely used in tanneries.

Principal market, London.

### b. *Raiidæ or Rays.*

*Ray oil* (*oleum rajæ; Rochenthran*, G.; *huile de raie*, F.) from the various species of ray. The liver of the ray is small and yields but little oil, but is nevertheless used for the purpose in Northern France, Belgium and Holland. Ray oil has a golden-yellow or pale-yellow color, smells and tastes less disagreeable than ordinary cod-liver oil, shows no acid reaction, and has a specific gravity of 0.928 at 59° F. Cold alcohol dissolves 1.5 per cent. and hot alcohol 14.5 per cent. of the oil. Stearin is separated near the freezing point. The oil contains more iodine than cod-liver oil. Its chemical composition is the same as that of other liver oils.

In connection with liver oils three oils are here mentioned which may eventually serve as a substitute for them.

1. *Eulachon, Oulachan, Oolachon or Oolacon oil or candle-fish oil*, is much used as a substitute for cod-liver oil. It is obtained from a fish called by the North American Indian *oulachan* or *candle fish* (*Thaleichthys pacificus*). It belongs to the smelt family. Vast shoals of these fish abound in the waters that lave the coasts of Alaska and British Columbia. So countless are their numbers that it is no uncommon sight in certain seasons of the year for the beaches to be covered with them for miles; they seem to be chased shoreward by large fishes, and throw themselves ashore in their fright, where they are dispatched and used as an article of food by the Indians. The oil tried out has certain nourishing and stimulating properties that fit it for a some classes of consumptives, being more easy of digestion than cod-liver oil.

Eulachon oil, as it is at present found in the market, contains much palmitin and probably also stearin, so that at the ordinary temperature it is only semi-liquid. Its specific gravity is 0.9071 at 59° F., and 0.912 at 77° F. The specific gravity of cod liver oil ranges from 0.92 to 0.93, it being generally 0.927 at 59° F.

When eulachon oil is mixed with sulphuric acid (1 volume acid, 5 volumes oil) the temperature rises to 131° F., but with cod-liver oil treated in the same manner to 233.6° F. The color reactions also differ from those of cod-liver oil.

On mixing eulachon oil with ⅓ its volume of *nitric acid* of 1.27 specific gravity, a rose color is immediately produced which slowly passes into amber-yellow. By standing 15 hours the mixture becomes considerably thicker and acquires a deep amber color.

Cod-liver oil treated in the same manner becomes first rose color, then rapidly amber-yellow, shows the same color after standing 15 hours, is more fluid than the former mixture, and is intermixed with partially thick layers separated one from the other.

*Sulphuric acid* does not produce with eulachon oil the same beautiful purple color as with cod-liver oil; it becomes deep brown and later on is inclined towards a yellow, as well as red, color. When eulachon oil is saponified the fatty acids separated therefrom amount to 95.85 per cent. of the oil.

The oil contains about 20 per cent. palmitic and stearic acids, 60 per cent. olein and 13 per cent. of a non-saponifiable substance, which being a peculiar and interesting body deserves most attention. At the ordinary summer temperature it exhibits an oily consistency, has a specific gravity much lower than oleic acid or any other constituent of ordinary fats, namely, 0.865 to 0.872 at 59° F., and appears to resemble the non-saponifiable constituent of sperm oil.

2. *Alligator oil and crocodile oil.* Several varieties of this oil are brought into commerce, and at present are chiefly used in tanning. Alligator oil is ointment-like, semi-fluid, of a reddish color, 0.928 specific gravity, and contains about 60 per cent. olein, 32 per cent. margarin and stearin, 1.5 per cent. free oleic acid, as well as 0.02 per cent. iodine.

Crocodile oil is of a reddish color, but more fluid than alligator oil. A mixture of both oils is also said to occur in commerce.

*Turtle oil.* In the Seychelles and Jamaica an oil obtained from turtles is used as a substitute for cod-liver oil. In both places yellowish, thick, fine-grained oils not unlike solidified olive oil are obtained, which become liquid by moderate heating, possess but little odor or taste and do not turn rancid.

Turtle oil, or more correctly "turtle egg oil" is produced by crushing the turtle eggs in an empty canoe with a wooden fork-like instrument, pouring water upon the mass and exposing it for some hours to the heat of the sun, when the oil separates on the surface.

Six thousand turtle eggs yield only 5 gallons of oil. At least 50,000 gallons of oil are annually brought from the Orinoco, the Amazon and the Rio, to Para, and 60,000 gallons are consumed by the tribes preparing it, so that from these figures it can be seen that though the turtles year after year visit the same sand-banks and each lays during the season three times 120 eggs, the issue of a large number of turtles is involved.

The eggs of various turtles, especially of *Padocnemis expansa* are used.

The shores of the Seychelles are visited chiefly by two species of turtles, *Chelonia mydas*, the green or gigantic turtle, and *Chelonia imbricata*, the hawk's bill turtle. According to J. J. Sharp, only the first-named species is used for the preparation of oil, which, according to J. H. Brooks, is there not obtained from the eggs, but from the fat of the animals by boiling. The yield is more abundant, a single turtle frequently furnishing 10 gallons of oil.

In Jamaica the oil is also obtained from the flesh of the turtle, probably from *Chelonia cahouana.*

The liver oils all show, on account of their content of gall constituents, the same behavior towards reagents, and are, therefore, readily distinguished from other train and fish oils.

*Fuming nitric acid* colors the liver oils, first, blood red, and then, blue-red to brown.

*Sulphuric acid* of 1.65 to 1.70 specific gravity causes violet to violet-black coloring.

*A mixture of sulphuric and nitric acids* produces first a yellow-red, then brick red, and finally a red-brown color with a violet hue.

*Soda lye and phosphoric acid* give a red color.

Liver oils are distinguished from each other by their specific gravity, and the cod-liver oils from ray oils by the first being blackened by chlorine. Their adulteration with other oils will be given at the end of this chapter.

### D. FISH OILS, ACTUALLY WASTE OILS.

1. *Herring oil* (*Häringsthran*, G.; *huile de hareng*, F.) from *Clupea harengus*, Linn., the common herring and *Clupea pontica*, the Astrachan herring.

2. *Sprat oil* (*Sprottenthran*, G.; *huile de spratt*, F.) from *Clupea spratta*. Cuv. = *Harengula sprattus*, Bl., the sprat.

3. *Sardine oil* (*Sardinenthran*, G.; *huile de sardine*, F.) from *Clupea sardinus*, Linn., the sardine.

4. *Pilchard oil* (*Pilchardthran*, G.; *huile de pilchard*, F.) from *Clupea pilchardus*, Bl., the pilchard.

5. *Anchovy oil* or *sardel oil* (*Sardellenthran*, G.; *huile d'anchois*, F.) from *Engraulis enerasicholus*, Cuv., the anchovy.

*Japanese anchovy oil* was, in 1885, for the first time brought to Europe, but did not meet with any favor. However, according to Villon, it is destined to play an important part in tanning, as well as in the manufacture of stearin and of soap. It contains 70 per cent. liquid oil, 30 per cent. fat, and is of the consistency of butter. Comparatively small quantities are at present exported, but the possibility of regular production exists.

6. *Menhaden oil* (*Menhadenthran*, G.; *huile de menhaden*, F.) from *Alosa menhaden*, Cuv., the menhaden.

The above oils are known either under the names mentioned or are designated in commerce according to the countries from which they are exported. Thus, for instance,

Swedish fish oil = Herring oil,
Russian fish oil = Astrachan herring oil,
Italian fish oil = Sardine oil,
Spanish fish oil = Pilchard oil,
American fish oil = Menhaden oil.

All the fishes mentioned in the foregoing, as well as allied species, form an important article of food and occur in commerce prepared in various forms. The waste obtained in preparing the fish is worked into oil, considerable quantities being thus obtained, which form a productive article of commerce.

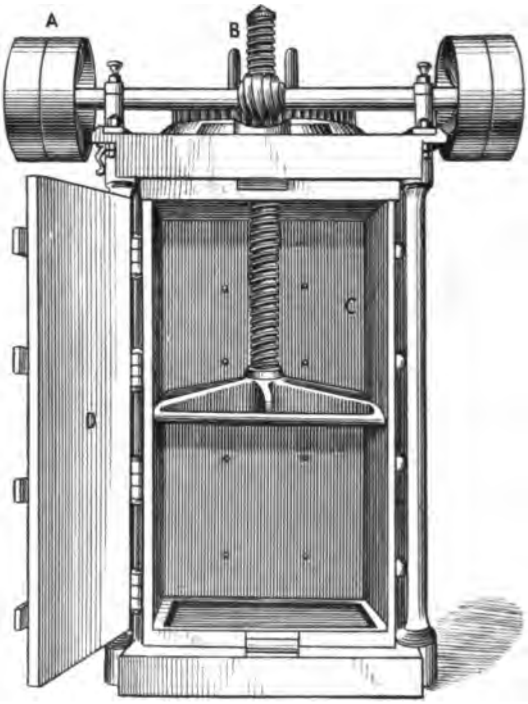
Before salting the herrings, sardines, pilchards, etc., the throats are cut, and the entrails drawn out. The throats, heads, and entrails, together with such entire herrings, sardines, etc., as cannot be quickly enough salted on account of the magnitude of the catch, are then boiled with water in large iron boilers, and the oil floating on the surface dipped out, and clarified in large vats. The residue is used in the manufacture of fish guano. It is more advantageous to boil the fish with sulphuric or hydrochloric acid.

The offal from other fishes, such as the salmon, sturgeon, etc., is worked into oil in a similar manner.

According to another method the fish are sprinkled with 5 per cent. of their weight of solution of chloride of iron or sulphate of iron at 45° Bé., which preserves them unchanged for 3 or 4 days. They are then converted into a pulp and pressed, whereby a large quantity of water and oil is obtained. The residual press-cake dries readily, becomes friable and can be pulverized. By expressing between heated metallic plates or by extraction with benzine or carbon disulphide further quantities of the fatty substance may be obtained. The residue is used as a fertilizer.

Fig. 230 exhibits a press for the expression of fish-waste, according to Rose, Downs and Thompson. Its construction will be readily understood from the illustration. The shaft *B* driving the screw is provided on each end with a fast and loose pulley, thus rendering possible the up and down moving of the press-plate without opening the press-space. The press-space itself is surrounded by a wrought-iron steamjacket. Expression is effected between metallic plates.

FIG. 230.



FISH-OIL PRESS.

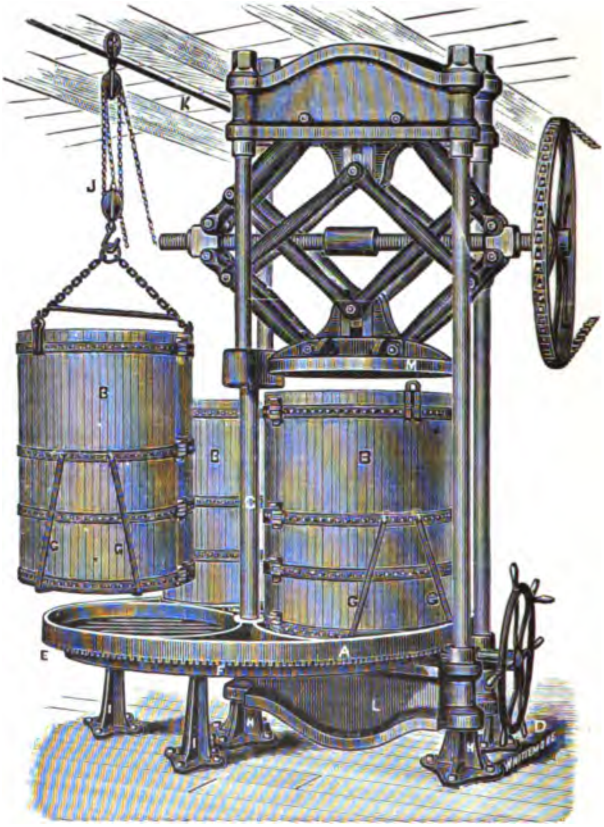
*Menhaden oil* is manufactured on a large scale in the United States. As a source of oil the menhaden is of more importance than any other marine animal. Its annual yield usually exceeds that of the whale (from the American fisheries) by about 200,000 gallons. In 1880 the number of persons employed in the entire industry in America was placed at 3635; the amount of capital invested $2,362,841; the value of products $2,116,787, including 2,066,396 gallons of oil, worth $733,424, and 68,904 tons of guano, worth $1,301,217. The refuse of the oil factories supplies a material of much value for manures. As a base for nitrogen it enters largely into the composition of most of the manufactured fertilizers. The amount of nitrogen derived from this source has been estimated to be equivalent to that contained in 60,000,000 pounds of Peruvian guano, the gold value of which would not have been far from $1,920,000. The yield of the menhaden fishery in pounds is probably triple that of any other carried on by the fisherman of the United States.

The manufacture of menhaden oil has been prosecuted for a few years only. The fish are caught on the coast of Maine in three different modes: 1st, weirs; 2d, gill nets; 3d, purse seines.

The primitive mode of making oil was by chopping the fish, boiling them in a kettle, and skimming off the oil that rose to the surface. A large percentage was by this process left in the scrap. By and by screw presses were introduced to press out the oil from the scrap. These were just like cider presses, and the fish were laid up, like apple pomace, in cheeses, but a coarse cloth was used for an envelope instead of straw. This mode is a very efficient one and is still in use in all except the larger class of establishments. The process of oil making in the larger works is essentially as follows: The fish are conveyed to the upper floor of the factory on wooden tramways in cars containing about twenty barrels each, and are dumped into large reservoirs from which the cooking tanks are replenished from time to time, or are emptied directly into the cooking tanks, which are filled to the depth of six inches with sea-water. From fifty to seventy-five barrels are placed in each cooking tank and then steam is turned on, and they are boiled for half an hour or more. In this way about two-thirds of the oil is separated; the remainder is expressed by means of presses. A press much used for the purpose, manufactured by G. H. Bushnell Company, of Thompsonville, Conn., is shown in Fig. 231. It is arranged for doing the largest amount of work in the shortest time. It is provided with a revolving table large enough to hold three curbs holding over three hundred gallons each, While one curb is under pressure, one may be filled and the other emptied. As soon as pressure is off curb No. 1, the table is revolved so as to bring curb No. 2 under the press, and so on.

The revolving table *A* holds three curbs *B B B*. Column *C* forms a centre for the table, which is revolved by a pinion gear on shaft, with hand wheel *D*, that engages in rack *E*. *F* is a trough passing around under the table to receive the liquid, and discharges it back of the press. *G G* are hooks to hold up

FIG. 231.



BUSHNELL CO. PRESS WITH REVOLVING TABLE.

bottom of curb, which is hinged to opposite side. *H H H H* are heavy supports for press to stand on. *I I* are stands to prevent the table from tipping if loaded heavily outside of press. *J* a pair of chain-falls attached to a truck running on track *K*, to raise curb up from table and traverse it to any convenient place to empty it. *L* is bed of press, where the revolving table rests when under pressure. *M* is a follower which passes down inside of curb.

The oil mixed with water running off from the press is carried into the "drawing-off tanks" while it is still hot, the water separating and sinking to the bottom. Great pains must be taken to separate the oil from the water before the impurities contained in the latter begin to ferment, for if this happens the quality of the oil suffers much. Moreover, in what appears at first to be pure oil there is a variable amount of finely divided fleshy substance that must be allowed to settle, as it will after a while, and the clarified oil drawn off before putrefaction sets in. In order to effect the separation the oil is commonly passed through a number of settling vats and a portion of the impurities deposited in each, and finally, before barreling, the oil is pumped into bleaching tanks, where it becomes clearer and whiter in the rays of the sun, and after one or two weeks' exposure is ready for shipment. If all these processes are successfully carried through, the oil is light-colored, sweet, and of prime quality; but, if it is exposed at any time to the influence of putrefying animal matter, it becomes dark and strong. The very strongest oil is made from the "*gurry*" or settlings of the oil, after fermentation, by steaming or boiling it over.

It naturally happens that every manufacturer makes several grades of oil, of very different quality, of which the best is very sweet, fine oil, bringing ten cents per gallon more than a strong article. Notwithstanding this fact, it is said to be the common practice of dealers to pour all grades into the same vat, and this has led manufacturers to take less pains to keep them separate.

It is a curious fact that oil made from early fish is not so good as that made later. It is called "weak" and brings in market five cents per gallon less. Gurry oil is sold for ⅓ less than the other grades.

Floating factories are in use, chiefly in Long Island Sound, in whose protected waters they operate to great advantage. They are now going out of use on account of the introduction of steamers. They are usually built upon the hull of some old vessel and are towed from point to point, gathering the fish from the smacks and working them into oil and guano as they move. Some of them are fitted up with machinery for very extensive manufacture. Two important objects are attained by the owners of floating factories: the objection to their business arising from the offensive odor is to a considerable extent removed; by following the movements of the fish time and expense are saved, for, bringing the factory to the fish, they obviate the necessity of having a fleet of lighters to carry the fish to the factory, which might often require two or three days.

In the following we give an account of an improved process of utilizing the scrap, devised by Mr. S. L. Goodale.

As now generally managed, the scrap remains in large heaps until shipped, in autumn or winter, to the points of manufacture into, or incorporation with, superphosphate. In this time a portion of the oil and water leaks away, so as to leave about 10 to 15 per cent. of the former and 48 to 53 per cent. of the latter. The elimination of the water is an advantage, but the specified per cent. of oil is lost, and a portion of the nitrogen is also lost, resulting from the partial decomposition of the mass, the formation and escape of ammonia. It were better if practicable, to drive off the water at once upon withdrawal from the press, so as to prevent the loss in question.

What has hitherto prevented the driving off of the water immediately by artificial heat has been the presence of so much oil, together with the gelatinous or gluey mass which is developed during the cooking, chiefly from the skins and bones. These render the process of drying the scrap a very difficult and tedious one, so much so that comparatively little has been put into market in that desirable form. The recent discovery of an easy and simple process for removing the larger part of the oil, and also at the same time the gelatinous hindrance to drying, gives promise of a speedy change in this respect.

While pursuing investigations relative to utilizing the menhaden as a source of concentrated food, Mr. S. L. Goodale found, by thoroughly washing the scrap as it came from the press with sufficient hot water, and agitation, that the oil globules were liberated from their entanglements in the fleshy tissues, and also from the creamy mixture with the gluey matter into which they were forced by the pressing, so that the greater part of it could be recovered by draining and re-pressing; and also that after such washing the scrap would bear heavier pressure than at first without "squirting." By this easy proces the oil product is largely increased, the scrap is left free from the gluey hindrance to drying, and contains less water to be dried out.

It may appear strange that so simple a method should not have been discovered sooner, but such is the fact. Work had been done on both sides of it. Re-pressing had been tried, using extra strong curbs, with very powerful pressure, but it failed to give satisfactory results. Re-cooking had been resorted to, which resulted in injury to the oil and in the development of an additional amount of the gelatinous matter. It is now seen that a simple thorough washing in hot water accomplishes the desired end with neither of these objectionable results. Scrap made by this process and dried in the open air was analyzed at the agricultural experiment station of Connecticut, and the statement of the director, Professor S. W. Johnson, of New Haven, shows the proportion of moisture to be reduced to 11.45 per cent., or about one-fifth that contained in the scrap fresh from the press, and the proportion of oil to 4.65 per cent., thus proving that the content of oil in the *washed scrap as it came from the press* (before drying it) had been reduced to less than 2½ per cent. According to these figures, the proportion of oil hitherto lost is by the new process reduced from an average of, say, 15 per cent. of the weight of the scrap as it commonly issues from the press, to about 2 per cent.; the balance, say 12 or 13 per cent., is saved. Let it be assumed, however, that only 10 per cent. can be realized in practice, and that the annual out-put of scrap from the factories of the Maine Association is only 40,000,000 pounds, this would give an annual saving of 4,000,000 pounds of oil, or 533,000 gallons, worth, at 40 cents per gallon, $213,200.

Other methods of extracting the oil from fish scrap have been proposed. They involve the use of fumes of benzine or carbon disulphide, which are brought into contact with the fish in air-tight chambers. The oil is absorbed by these substances and collects in tanks in the floors of the chambers. Any surplus of benzine or carbon disulphide which may remain in the oil is expelled by distillation.

The *naphtha* process for extracting the oil consists in subjecting the scrap, in an inclined iron cylinder, to the action of vapors of naphtha, which combine with the oil, and the latter, in a state of solution, filters away at the lower end of the cylinder. The naphtha is then removed by evaporation. The process is slow, costly, and dangerous.

The uses of menhaden oil are manifold. It is chiefly employed as a substitute for the more costly and popular oils, and to adulterate them. It is sold largely to tanneries for currying leather. After the hide has been dressed, *i. e.*, after its coarser fleshy parts have been pared off, the oil mixed with tallow is applied. This is technically called "stuffing," and results in qualifying any residue of alkali left from the liming process, and in filling the pores and softening the leather.

A considerable quantity of the oil is used as a burning oil in coal mines to fill the small lamps, one of which is fastened to the cap of each miner. It is then mixed with paraffin or some of the heavier oils. Some is also sold to be used in the manufacture of rope. A small quantity is used annually for lubricating purposes, but, on account of its gummy nature, it is not much in favor among machinists. It is used in adulterating linseed oil, and is also sold as a substitute, its cheapness and durability rendering it specially valuable for rough outside work and for painting ships. Mixed with other oils it is found to be very serviceable for the painting of interiors, and its use is attended with decided economy, its price being about one-half that of the best linseed oil. Some of the most pure is said to be put into the market as olive oil.

Most of that which is exported is used in the manufacture of soap and for smearing sheep after they have been sheared, to keep off ticks.

Several grades of menhaden oil are recognized. The "Oil, Paint, and Drug Reporter" usually quotes under the heads of "select light strained," "select light," "choice brown" and "inferior to dark," and "gurry."

The oil and guano factories are located chiefly on the coasts of Maine, Rhode Island, Connecticut, and Long Island, a few being also found on the coast of New Jersey, on Chesapeake Bay, and on the Southern coast.

The principle market is in Boston and New York; some is also sold in New Bedford, and considerable quantities are shipped to London, Liverpool, and Havre direct.

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The fish oils possess a specific fishy odor and taste and occur in commerce of a pale yellow to brown color. Their specific gravity varies from 0.925 to 0.930 at 59° F. Stearin is deposited near the freezing point. Cold alcohol absorbs only 2 per cent. and hot alcohol 3.5 per cent. Five volumes of fish oil require two volumes of ether for solution.

*Fuming nitric acid* colors all fish oils brown.

*Sulphuric acid* of 1.65 to 1.70 specific gravity gives first a characteristic greenish color, which later on becomes brown, and finally entirely black.

*A mixture of equal volumes of sulphuric and nitric acids* produces first a yellow, then a greenish, and later on a brown color.

The behavior of fish oils towards *soda lye* and *phosphoric acid* is the same as that of other train oils.

In testing train oils the use of the tongue as a reagent is not very agreeable, but, with some experience, very safe, for all the oils have a peculiar taste and odor which cannot be described, but which, experienced several times, are not readily forgotten.

The better qualities of train oil are generally only adulterated with those of less value, and a sophistication with other oils occurs perhaps only in cod-liver oil for medicinal purposes.

In making the test, the specific gravity is to be taken into consideration. Shark oil is the lightest, with a specific gravity of 0.890; next come the whale oils, with one of 0.910 to 0.925; then the seal oils, with 0.915 to 0.930; liver oils, with 0.920 to 0.925; fish oils, with 0.925 to 0.930. These statements refer only to the pale and light brown oils; the brown and dark oils are on average 0.005 heavier.

For the recognition of *genuine cod liver oil*, Meyer recommends shaking 10 parts of oil with 1 part of a mixture of equal parts of concentrated sulphuric and nitric acids in a stoppered glass bottle, whereby genuine cod-liver oil acquires a fiery rose color which quickly passes into lemon-yellow, while with other train oils the transition to yellow is not so pure but more brownish-violet.

On agitating train oil with concentrated *aqua regia* a greenish dark yellow liniment is formed with *genuine cod-liver oil* which turns brown in half an hour and remains so, while white seal oil or a mixture of it with genuine cod-liver oil gives a pale yellow, slightly shaded mixture.

More suitable for the distinction of the separate varieties of train oils is their behavior towards fuming nitric acid of 1.50 specific gravity.

By bringing 10 to 15 drops of the oil to be examined upon a watch crystal and allowing to run down on one side 3 to 5 drops of fuming nitric acid the different train oils behave as follows:

*Cod-liver oil*, on the point of contact becomes red and after stirring with a glass rod, fiery rose color, which in a short time, however, passes into a pure lemon-yellow.

*Coal-fish oil* on the place of contact with fuming nitric acid becomes intensely blue and on stirring passes into brown, which lasts two or three hours finally passing into a more or less pure yellow.

*Japanese anchovy oil* behaves in a similar manner, though sometimes red lines besides the blue are formed.

*Seal oil* treated in the above described manner with fuming nitric acid shows at first no change of color and only after some little time is colored brown.

Adulterations with other oils occur chiefly in liver oils. According to E. Salkowsky the following data may be utilized for recognizing such adulterations.

*The solidifying and fusing points.* With liver oils the solidifying point is quite low, though the different kinds show considerable variations in this respect, which may be traced to the fact that in some varieties the portions fusing with difficulty are already separated by chilling. The time required for solidifying is also of importance, it being possible to recognize adulteration with some oils, such as palm oil, cocoanut oil, palmkernel oil, of which up to 20 per cent. have been added, by the rapid solidification of the liver oil.

Reichert-Meissl's number (see butter) for liver oils was found to be 0.1 to 0.2 after deducting a correction of 0.2 (for the indicator). But slightly higher numbers were found for most fat oils, only those for cocoanut oil and palmkernel oil being 7.38 and 3.48.

*Reaction with sulphuric acid* (content of phytosterin). The test is most suitably made in two ways, on the one hand, by directly adding sulphuric acid to the liver oil in a watch-crystal, and, on the other, by dissolving a few drops of oil in chloroform, adding sulphuric acid and shaking; the result is a blue color. This coloration is not caused by cholesterin, of which up to 3 per cent. is present, but is due rather to a coloring matter called *lipochrome.* Of animal fats, egg-fat, and to a lesser degree, butter-fat especially show a content of lipochromes, while of vegetable fats the orange-colored palm fat is distinguished by a large content of lipochromes. The cholesterin of liver oil and that of vegetable oils are not identical, the latter corresponding to phytosterin (described by Hesse). Cholesterin crystallizes to a paste of laminae, and phytosterin to solid needles grouped in bunches. The fusing point of phytosterin lies between 269.6° and 273.2° F., and that of cholesterin at 294.8° F. These differences are sufficiently characteristic to allow of the determination of vegetable fats in liver oils.

Saponify 10 grammes of liver oil with 10 grammes of potassium hydrate and a small quantity of alcohol. Dissolve the resulting soap in sufficient water to make 600 or 700 cubic centimeters, shake with ether (about 500 c.c.), filter the ethereal extract, evaporate and eventually purify by again saponifying with alcoholic potash lye, when cholesterin of sufficient purity is obtained. Cholesterin from pure liver oil fuses at 294.8° F., and that of oil mixed with 20 per cent. vegetable oil at 282.2° to 284° F. Adulteration may also be established by a microscopic examination of the cholesterin.

Good varieties of liver oil contain but a small quantity of *free fatty acids*, about 0.25 to 0.69 per cent., while most commercial vegetable oils contain more.

The determination of the volatile fatty acids insoluble in water, mentioned by Salkowsky, is quite a difficult method, uncertain in its final results and therefore not available for the practice.

Finally, the intentional *mixing of linseed oil with fish oil* deserves attention. For its establishment chlorine is generally conducted into the mixture. However, in practice, irregularities occur which may lead to errors.

*The action of chlorine upon pure linseed oil* is, according to A. Ruffin, as follows: 1. Action of a current of chlorine upon linseed oil kept in a closed bottle to prevent the access of air. The oil acquires a green color, which turns brown on interrupting the current of chlorine; the brown color appearing more slowly without the access of air. Duration of the reaction, 24 hours. By the further admission of chlorine the color becomes black. 2. Action of chlorine upon linseed oil in the presence of air. The oil becomes first green, then black. Duration of the reaction, 6 hours. 3. Action of chlorine upon mixtures of fish oil and linseed oil. The oil becomes immediately black without being previously colored green. Hence, in making the test it has first to be ascertained whether the oil is fresh or old, whether it has been kept in closed or open vessels, and finally a comparison with pure oil has to be made.

From liver oil to which iodine has been artificially added the latter can be extracted by agitating with a little rectified spirit and then testing the last for iodine, while the determination of the iodine in genuine liver oil has to be conducted in the manner given on page 61.

### 4. *Glycerides of Palmitic and Stearic Acids, etc., or Fats.*

#### A. Solid Fats of the Vegetable Kingdom.

#### *Simarubeæ (Terebinthinæ).*

*Dika butter, oba oil, or wild mango oil* (*Dikabutter*, G.; *huile de dika*, F.) is obtained from *Irvingia Barteri*, Hock., a tree indigenous to the west coast of Africa and called by the natives the wild mango. The seed contains 60 to 65 per cent. of fat, which is obtained by boiling the seed in water and pressing. This fat is the dika butter. When fresh it has a pleasant mild taste and odor, is white, and keeps quite a long time. By long storing it becomes yellow and rancid. It has a specific gravity of 0.820 at 59° F., melts at 86° to 89.5° F., and contains, besides glycerides of lauric acid and myristic acid and a very little palmitic acid, a few crystals of free fatty acids.

Dika butter is readily saponified. It is used in the manufacture of soap and candles, and frequently serves as a substitute for cocoanut oil.

| | |
|---|---|
| Saponification number of dika butter ..... .............. | 173 |
| Iodine number of dika butter ........................ | 30.9–31.3 |

### *Burseraceæ* (*Terebinthinæ*).

***Java almond oil*** (***Java Mandelöl***, G.; ***huile de canaria***, F.) is obtained from the nuts of ***Canarium commune***, Linn., or ***Colophonia mauritiana***, D. C., a tree indigenous to the Moluccas and Malabar, and cultivated in tropical Asia. The kernels of the nuts are eaten like almonds and yield 40 per cent. of a semi-solid fat of an agreeable and sweet taste. It keeps very long without turning rancid, and is used as a fine table oil.

### *Euphorbiaceæ* (*Tricoccæ*).

*Chinese vegetable tallow* (***oleum stillingiæ***, ***Stillingiatalg***, G.; ***suif végétal de la Chine***, F.) is obtained from the seeds of ***Stillingia sebifera***, Juss., or the Chinese tallow tree. The fruit of the tree is a black, somewhat globular capsule, and contains three egg-shaped seeds coated with a thick, hard, tallowy layer, while the kernel contains a fluid fat. The seed yields 20 to 30 per cent. of tallow, which is obtained in the following manner: The seed, separated from the ripe capsules, is placed in large wooden cylinders, provided with numerous holes, and treated with steam, which causes the tallow to melt and run off. When cold the tallow is remelted and filtered. The seed is then comminuted, the resulting mass heated with some water and pressed, whereby a fluid oil called "*Ting yu*" is obtained which has some drying properties and is used in the manufacture of varnish and as lamp oil. Or the seed is comminuted in stone mortars, exposed to hot steam, and the mass pressed, the tallow obtained being considerably softer.

The tallow obtained according to the first method is pure white or slightly greenish-white, inodorous, quite hard, and spread upon paper causes but few grease spots. It has a specific gravity of 0.918 at 59° F., melts at 112° F., and consists chiefly of glycerides of palmitic acid and very little stearic acid.

The tallow obtained by the second method forms a light, white mass of a slight odor and turns yellow to brown by storing. In consequence of admixed acetic and propionic acids, it shows a slight acid reaction. Its specific gravity is 0.915 at 59° F., it fuses at 95° F., solidfying again only at —7.6° F.

Large quantities of Chinese vegetable tallow are now used in England for the manufacture of candles, and it is also being gradually introduced on the Continent.

### *Rhizoboleæ* (*Malpighinæ*).

1. *Rhizobolus butyrosa*, W., the butternut tree, is of importance to the inhabitants of Brazil and Guiana, because besides the nuts which are of an agreeable taste the oleiferous, butyraceous middle layer of the pericarp is used like our butter for preparing food.

2. *Rhizobolus amygdalifera*, Aubl., = *Caryocar Brasiliensis*, Cuv., the almond-bearing caryocar, a tree 160 to 260 feet high, indigenous to Brazil, furnishes the *caryocar oil*, *huile de piguia*.

3. *Caryocar tomentosum*, Cuv., = *Pekea guianensis*. The nuts of an agreeable taste are brought into commerce as *sawarri* or *suari nuts*, yields a fat known as *suari butter*, *huile de noix de Sawarri*.

### *Sapindaceæ*) *Malpighinæ*).

1. *Soap-tree oil* (*oleum sapindi*; *Seifenbaumfett*, G.; *huile de savonnier*, F.) is obtained from the seed of *Sapindus emarginatus*, Roxb., or the soap tree, indigenous to Southern India, and cultivated in Bengal and the West Indies, and also recently in France. The dried berries are brought into commerce under the name of " soap berries." The fleshy part of the fruit is very rich in saponin; a small piece mixed with water giving a copious lather, which is used in India for washing, and in France for washing silk dyed with aniline colors.

No saponin is contained in the woody stone, seed, and husk, but the thick cotyledons contain about 30 per cent. of a white fat, which is obtained by extraction. It is semi-fluid at 68° F., and melts to a clear oil at 86° F.

Soap-tree oil has a somewhat characteristic odor. In India is used mechanically, and, as it readily saponifies, is also employed in the manufacture of soap.

2. *Rambutan tallow*, from the seed of *Nephelium lappaceum*, Linn., a tree indigenous to the Sunda Islands, Malacca, and some provinces of China. Rambutan tallow contains, besides a little oleic acid, almost pure arachidic acid. It melts at 149° F.

3. *Macassar oil*, which was formerly very celebrated for promoting the growth of the hair, is obtained from the seed of *Schleichera trijuga*, Willd., a tree of the East Indies. The seed is comminuted, pressed, or boiled in water, and the oil collecting on the surface carefully lifted off. Considerable quantities of the oil were formerly imported, but what at present comes into commerce under the name of "macassar oil" is mostly a mixture of cocoanut oil and ylang-ylang extract, colored red with alkannin. Genuine macassar oil is of a semi-fluid consistency at the ordinary temperature, of a yellowish-white appearance, and in odor slightly resembles oil of bitter almonds. The beneficial action of the oil is ascribed to the hydrocyanic acid which it is said to contain.

4. *Pekea tallow* is obtained from *Pekea butyrosa*, Aubl., a tree indigenous to Guiana, and *Pekea tarnatea*, Aubl., a tree of the Antilles.

### *Meliaceæ* (*Ampelideæ*).

1. *Andiroba fat, carapa oil or crab-wood oil* (*Carapafett*, G., *huile de carapa*, F.) is contained in the seed of *Carapa guianensis*, Aubl., and of *Carapa Moluccensis*, Lam., the first variety occurring in Brazil, Guiana, and West Africa, and the latter on the sea-coasts of India, Ceylon, the Moluccas, etc. The seed contains from 40 to 50 per cent. of tallow, which is obtained by comminuting or boiling the seed and pressing. It is used in France and England in the manufacture of soap. The natives use the fat for making the skin supple, and as a protection against the bites of insects.

Andiroba fat is colorless and almost solid; it melts at 73.5° to 77° F., and solidifies at 64.5° F. It has a strong, bitter taste, which, according to Cadet, is due to small quantities of strychnine, and can only be removed by repeated and continuous boiling in water. The odor of the oil is slightly acid and somewhat aromatic.

Alcohol dissolves the fat very sparingly. In ether it is completely and readily soluble. It saponifies quickly and entirely, as it consists chiefly of glycerides of palmitic acid, besides oleic and a little stearic acid. With the microscope fine needles of free crystals of fatty acid can be recognized.

2. *Nimb oil or Kohomba oil* (*Zedrachöl, Veppamfett,* G.; *huile de Veppam, huile de Margosa,* F.) from the seed of *Melia azedarach,* Linn., indigenous to Northern India and cultivated as an ornamental plant in Southern Europe and North America. The sed contains 50 to 60 per cent. of oil; it is comminuted and pressed. Nimb oil is also obtained from the seed of *Melia indica,* Brand, which contains only 40 to 50 per cent.

The oil is dark yellow, has a sharp, bitter taste and an acrid disagreeable leek odor. It is semi-fluid at above 59° F. It is used as lamp oil, in the manufacture of soap, and, internally, as a vermifuge.

3. *Mafura tallow* (*Mafuratalg,* G.; *suif de Mafura,* F.) from the fruit of *Mafueira oleifera,* Bert., a tree indigenous to Mozambique, in the region of the Zambesi, and the White Nile. The seed is very bitter and contains 60 to 65 per cent. of fat. It is comminuted and boiled with water, as but little fat can be obtained by pressure. The residue, which contains about 40 per cent. of nitrogen, is used as a fertilizer.

Mafura tallow is yellowish, tasteless, and of a cacao-like odor, It melts at 107.5° F., solidifies at 97° F., and has a radiated structure. It is readily soluble in ether, but very sparingly in hot alcohol, and consists chiefly of palmitin and some olein. It is used in the manufacture of soap.

The seed can be procured in quantities from Mozambique, Madagascar, etc.

### *Dipterocarpeæ* (*Columniferæ*).

1. *Piney tallow or Malabar tallow* (*Malabartalg,* G., *suif de*

*piney*, F.) is obtained from the seed of *Vateria indica*, Linn., or the piney tree, which is indigenous to Western India, Malabar, etc. The seed is roasted, ground, boiled with water, and the oil skimmed off.

Piney tallow is tasteless and of a somewhat fragrant odor. It is at first yellowish, but becomes pure white by storing for some time. As regards consistency, it is intermediate between wax and stearin. It melts at 98° F. and solidifies at 87° F. Its fracture is granular, radiated with exceedingly small needle-like crystals. Its specific gravity is 0.9150 at 59° F. It is readily saponified by alkalies, and colored green by chlorine. To alcohol it yields about 2 per cent. of an oil of an agreeable odor. It consists of 75 per cent. of palmitin, 25 per cent. of olein, and a quantity of crystallized free fatty acids. Its elementary composition is:

| | | |
|---|---|---|
| Carbon | 77.00 | per cent. |
| Hydrogen | 12.30 | " |
| Oxygen | 10.70 | " |
| | 100.00 | " |

| | |
|---|---|
| Saponification number | 191.9 |
| Fusing point of the fatty acids | 134° F. |
| Solidifying point of the fatty acids | 130.6° F. |

It is highly esteemed in the manufacture of candles, and is much used for that purpose in England. It has the advantage that the candles manufactured from it diffuse an agreeable odor, flicker but little, and consume the wick so that it need not be snuffed.

2. *Borneo tallow* (*Borneotalg*, G.; *Suif végétale de Borneo*, F.) is obtained from one or more trees of the genus *Hopea*, found in the south and east divisions of Borneo. The Dyaks call the fat *Kakawang* and the tree which yields it *Upu Kakawang*. This tree is one of the giants of the forest. The bark yields a red dye.

The preparation of the fat is very simple. When the ripe fruit falls on the ground it is allowed to germinate a little in a moist place. It is then dried in the sun until it becomes brittle. The fruit is then deprived of its shell and put into a rattan or bamboo basket suspended over boiling water. When it has been well steamed the fruit becomes soft and plastic like dough. The fat is then expressed by squeezing the doughy mass in a cloth, and is poured into joints of bamboo, by which it receives the cylindrical form in which it is met with in commerce. Some Dyak tribes press the fruit by means of two beams. But it is probable that by neither of these processes all the fat is obtained. The fat is also prepared in Java and Sumatra. By the natives the tallow is used for culinary and lighting purpases. It is largely exported to England for use as a lubricating agent. For this purpose it has proved most valuable, far surpassing even olive oil. In Manilla it has been employed in the manufacture of candles. The fat contains glycerin and about 95 per cent. of saponifiable matter which has less olein in it than animal fat.

3. *Niam fat*, from *Lophira elata*, Schiv., indigenous to East and West Africa.

### *Buettneriaceæ* (*Columniferæ*).

*Cacao butter* (*oleum theobromæ, oleum cacao; Cacao butter*, G.; *beurre de cacao*, F.) is obtained from the seeds or nibs of *Theobroma cacao*, Linn., or the cacao tree and various allied species. The habitat of the tree is Mexico and Demerara, and it is cultivated in the West Indies, Central America, Mauritius, and elsewhere. The seed, freed from the adhering pulp and dried in various ways, is called "unfermented cacao," while the better qualities are subjected to a kind of fermentation and are designated as "fermented cacao." The seed consists of about 12 per cent. of shell and 88 per cent. of kernel, the latter containing 45 to 55 per cent. of oil.

| | Peeled Caracas. | | Peeled Guayaquil. | |
|---|---|---|---|---|
| Fat | 52.20 p. c. | | 47.85 p. c. | |
| Organic substances | 39.00 " | | 41.50 " | |
| nitrogenous substances therein | | 17.40 p. c. | | 18.30 p. c. |
| Ash | 3.20 " | | 4.45 " | |
| Water | 5.60 " | | 6.20 " | |
| | 100.00 " | | 100.00 " | |

Among the organic substances is found about 1.5 per cent. of theobromine $C_7H_8N_4O_2$, the alkaloid of cacao seed, about 1 per cent. of which is also contained in the shell; further, 3 to 4 per cent. of cacao red and 7 to 8 per cent. of humic acid.

Formerly the roasted and ground seed was boiled with ten times the quantity of water, the oil skimmed off, and the residue pressed. But this primitive method has been abandoned, and at the present time the seed previously peeled and roasted is ground very fine, and the resulting mass, after heating to from 158° to 176° F., is packed in canvas bags and pressed between hot iron plates. The oil running off is filtered through a previously dried filter. The yield is about 30 to 35 per cent. The press residue, which contains from 10 to 15 per cent. of oil, is worked into chocolate. To obtain cacao powder almost free from oil, the roasted and ground seed is extracted with benzine or ether.

Cacao butter, fresh, is yellowish-white, but becomes pure white by age. It has a pleasant taste and odor of cacao. By boiling with alcohol it loses its odor and color. It is remarkable for keeping very long without becoming rancid. It is of the consistency of tallow, melts below 86° F., and some varieties even at 77° F., and solidifies at 69° F. Its specific gravity is 0.890 to 0.900 at 59° F. It is readily soluble in ether, chloroform, and oil of turpentine, and also in hot absolute alcohol, though after cooling ½ per cent. remains undissolved. Since it consists chiefly of glycerides of stearic and oleic acids, besides palmitic and arachidic acids, it is readily saponified. The mixture of fatty acids separated by acid melts at 124° F.

Cacao butter contains:—

| | |
|---|---|
| Carbon | 75.20 per cent. |
| Hydrogen | 11.90 " |
| Oxygen | 12.90 " |
| | 100.00 " |

The rather high price of cacao butter gives rise to many adulterations with wax, stearin, paraffin, and beef-kidney fat. For the determination of its genuineness, the purity of taste and odor, and the melting point, are of special importance. The melting point must not be below 77° F. and not over 86° F., that of cacao butter mixed with fat oils being below the first, and that of cacao butter adulterated with the above-mentioned substances above the latter.

For testing the purity of cacao butter, Dieterich has recommended an examination with the polarizing microscope. For this purpose fuse together equal parts of cacao butter and liquid paraffin, bring a drop of the mixture upon the object-bearer, gently press a glass cover down upon it and let it stand twelve hours at a temperature not exceeding 41° F. Twenty times magnified the cacao butter appears in the polarized light in form resembling palm fans and playing in all colors. An addition of 10 per cent. tallow separates it in nodules composed of needles, which with ox tallow show the polarizing cross, while the nodules of sheep tallow are colorless. Cacao butter adulterated with only 5 per cent. tallow gradually becomes white and acquires a disagreeable rancid odor. A small addition of tallow may still better be recognized by saturating a wick with the melted butter, igniting it, and blowing it out.

An adulteration of cacao butter with cocoanut oil may be recognized by treatment with various solvents. Cacao butter dissolved at a moderate heat in 3 parts of amyl-alcohol shows in the course of one day a crystalline sediment, amounting to about ¾ of the fat, and a clear supernatant solution; at a medium temperature cocoanut oil remains entirely in solution. Dissolved in 3 parts benzine cacao butter gives a

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turbid solution, which after 24 hours deposits about 5 per cent. of the fat and remains clear. With 3 parts of benzol and 3 parts of ether, both fats give at the ordinary temperature solutions which remain clear. Cacao butter dissolves in 5 parts of absolute alcohol heated to boiling, and cocoanut oil in 2 parts at 140° F. and in 5 parts at 86° F. Cacao butter dissolved in 10 parts alcohol separates on cooling small-grained crystals which fill up the layer of fluid about one-third; cocoanut oil, on the other hand, when dissolved in alcohol, yields a fluid which at a medium temperature remains liquid for several hours, when it separates in acicular crystals adhering together in spherical balls. A mixture of both fats is separated by treatment with 90 per cent. alcohol at 122° to 140° F., and the constituents are separately examined.

An adulteration with paraffin makes cacao butter feel soapy and decreases its specific gravity. An addition of stearin is recognized by the higher fusing point, and by boiling with dilute soda solution, sodium stearate being formed, which, on compounding with sulphuric acid, separates stearic acid.

| | |
|---|---|
| Saponification number of cacao butter | 192–202 |
| Iodine number of cacao butter | 32–37.7 |
| Fusing point of the fatty acids | 125.6° F. |
| Solidifying point of the fatty acids | 123.8° F. |

Cacao butter is obtained as a by-product in all large chocolate factories, and is used in the manufacture of perfumery, cosmetics and soap, as well as in medicine as an emollient and as a basis for suppositories and pessaries.

### *Clusiaceæ* (*Guttiferæ*).

1. *Goa butter, cokum butter, or concrete oil of mangosteen* (*oleum garciniæ; Kokum butter*, G.; *beurre de cocum*, F.) is contained in the seed of *Garcinia indica*, Chois., or the mangostan, a tree of the East Indies. The fruit contains a red marrow, the red juice of which has an agreeable but very acid taste. It was already known to the Portuguese in 1563, who

used the juice for dyeing and in the preparation of vinegar. Up to 1830 nothing was known about the fat contained in the seed, but since that time it has been used for the adulteration of "*ghee*," the clarified butter of Hindostan.

The seed is comminuted and boiled with water, and the fat which is collected, after cooling, is clarified by remelting and filtering. The yield is only about 10 per cent., though the seed contains considerably more fat.

Goa butter is white or pale gray-yellow, solid at ordinary temperature, and friable. It has a slight but not disagreeable odor, and a mild, oily taste. By long storing it becomes rancid, and acquires a brownish color, with an efflorescence of crystals. It melts at 104° to 106° F. to a clear, straw-colored liquid, which solidifies at 89.5° F., whereby it becomes heated to 97° F. In ether it is readily soluble, but only sparingly so in alcohol. By saponification with soda lye it yields a hard soap, and, after decomposing the latter with sulphuric acid, nearly the same quantity of fatty acid as that of the butter used is obtained.

Goa butter consists of the glycerides of stearic, myristic, and oleic acids, besides free fatty acids; volatile fatty acids, such as butyric and valerianic acid, are not present. It is chiefly used for adulterating butter, and in medicine as a basis for suppositories and pessaries, in fact as a substitute for cacao butter and spermaceti. Rancid Goa butter is utilized in the manufacture of soap.

2. *Gamboge butter* (*Gambogebutter*, G.; *suif de Gamboge*, F.) from *Garcinia pictoria*, Roxb., a tree of the East Indies. The seed is worked in the same manner as the foregoing and yields a yellow fat, which is used as butter by the poorer classes, and as burning oil by the rich.

3. *Sierra Leone butter, or African vegetable butter* (*Afrikanische Pflanzenbutter; beurre ou suif de Sierra Leone*, F.) from *Pendadesma butyracea*, Don., or the African butter tree. The seed comminuted and boiled with water yields 32 per cent. of a pale yellow fat, which melts at from 97.8° to 98.5° F.

4. *Poonseed oil* (*Tacamahacfett*, G.; *huile de Tamann*, F.) from *Calophyllum inophyllum*, Linn., or the tacamahac, a native of the Malayan Archipelago and the East Indies. The seed yields 60 per cent. of a dark green fat, which is solid at 50° F. and semi-liquid at higher temperatures. It has an aromatic odor and taste, and a specific gravity of 0.950 at 59° F. It is used in the manufacture of soap.

5. *Calaba oil* (*Calabafett*, G.; *huile de Calaba*, F.) from the seed of *Calophyllum calaba*, Br., a tree of the Antilles. It is used as lamp oil and in the manufacture of soap.

6. *Mesua ferrea*, Linn., the Ceylon ironwood tree or Nagkasur.

### *Bixineæ* (*Cistifloræ*).

*Chaulmoogra oil or gynocard oil*, (*Chaulmugraöl*, G.; *huile de chalmogree*, F.) from *Hydnocarpus odoratus*, Lindl., a large tree found in the forests of the Himalaya, in the East Indies and Malayan Islands. The seed, called in China "*tafungtse*," is officinal in England, and yields by expression over 30 per cent. of oil, and extracted with ether 50 to 52 per cent. Chaulmoogra or gynocard oil, when not entirely fresh or pressed from old seed, has a characteristic (bird-glue like) odor and taste, and is somewhat brownish; otherwise it is hard and without taste or odor. The expressed fat is hard up to 62.5° F., the extracted only to 57° F. In India, China, and Siam the oil is used for many purposes, but specially as a remedy for rheumatic gout, and various diseases of the skin.

### *Polygalaceæ*.

*Polygala butyracea*, a bushy plant indigenous to Timoré and Koranka, in the region of the Upper Niger. The fruit is an elliptical, four-sided, smooth capsule. The seed is egg-shaped, 5 millimeters long and 3 millimeters wide, and shows a quite loose oleiferous endosperm, which covers the embryo everywhere, the cotyledons of which are comparatively large and its radicles short and curved towards the hilum. The oily kernel consists of numerous thin-walled cells arranged in horizontal layers, which are completely filled with roundish solid granules of fat, and contain no gluten. The fat obtained from the seed is called *maloukang butter.* It tastes like nut-kernel and looks like yellowish butter. Fusing point 125.6° F.; solidifying point 91.4° F.; specific gravity 0.94. With concentrated sulphuric acid the fat acquires an orange-red, and later on a brown color. It readily saponifies with alkalies and contains 31.5 per cent. olein, 4.8 per cent. free palmitic acid, 57.54 per cent. palmitin, and 6.16 per cent. myristin, besides a small quantity of formic and acetic acids.

In the country of its origin *maloukang butter* serves for seasoning food; or the comminuted kernels are directly mixed with maize or rice.

### *Myristiceæ* (*Trisepalæ*).

1. *Nutmeg butter* or *expressed oil of nutmeg* (*oleum myristicæ expressum, oleum nucistæ; Muskatbutter*, G.; *beure de mucade*, F.) is obtained from the seeds of *Myristica officinalis*, Linn. = *Myristica moschata*, Thunb. = *Myristica fragrans*, Houttuyn, or the common nutmeg, which is indigenous to the Moluccas, and cultivated in many tropical countries, the East Indies, West Indies, Brazil, etc. Nutmeg butter is manufactured by heating broken and imperfect nutmegs (sound ones being too dear for the purpose) to a pulp, which is inclosed in a bag, exposed to the vapor of hot water, and then pressed between heated iron plates. The yield is about 20 to 23 per cent., seldom more. Fusing point of the butter, 123.8° F.

Nutmeg buttter is of the consistency of tallow, but more friable, orange-colored, and of a fragrant and spicy taste and odor. It has a specific gravity of 0.990, and is soluble in 2 parts of warm ether and 4 parts of warm alcohol. Cold alcohol dissolves only the coloring substance, volatile oil, and fluid fat, of which, together with 40 to 45 per cent. of fat, the butter is composed. It consists chiefly of the glyceride of myristic acid, and contains besides 6 to 8 per cent. of the volatile oil of nutmeg.

The test for adulterations is based upon its behavior towards hot ether and alcohol, the solution, in the presence of other fats, being not entirely clear. Tallow is detected by the disagreeable tallow-odor diffused by igniting and extinguishing paper soaked in the suspected sample.

| | |
|---|---|
| Acid number | 22.4 |
| Ester number | 134.4–137.2 |
| Saponification number | 154–159.6 |

Nutmeg butter was formerly exclusively brought into commerce, by way of Holland, in oblong cakes having nearly the form of common brick, but somewhat smaller and packed in monocotyledonous leaves commonly called "flag leaves." At the present time much of the oil is manufactured in Europe, which is put up in the same shape but packed in paper. When discolored and hardened by age the oil is called "*Banda soap*."

Nutmeg butter is occasionally employed externally in chronic rheumatism and palsy, but its chief use is in perfumery.

2. *Otoba butter* (*Otobafett*, G.; *suif d'Otoba*, F.), obtained by expression from the fruit of *Myristica otoba*, Humb., or the Otoba nutmeg, a native of New Granada. It is of a slightly yellow color, of the consistency of nutmeg butter, and when fresh has the same odor, which, however, is changed to a disagreeable one on melting. With age it acquires a light brown to dirty brown color. It melts at 100.5° F., and consists of myristin and olein.

3. *Becuiba tallow* (*Becuhybafett*, G.; *beurre d'Ucu-uba*, F., from *Myristica bicuhyba*, Schott. The seed has a black, fragile testa with broad furrows, and a kernel resembling nutmegs in form, structure, and size, and having an agreeable, cacao-like odor and a taste resembling that of cacao butter, somewhat suet-like, and with a bitter after-taste. The crushed seed, subjected to hydraulic pressure, yields 47.56 per cent. of fat. The fat extracted with ether is light yellow, and its ethereal solution yields shining, white, scaly crystals and a yellow oily mother liquor. The expressed fat is yellowish-brown, and the surface

becomes covered with a white crystalline efflorescence. Melted, it is dark brown and solidifies with a wavy surface. The fat melts at 108.5° to 109.5° F., and solidifies at 89.5° to 90.5° F. It is readily soluble in hot ether, carbon disulphide, and chloroform, and partly soluble in hot alcohol. It consists of the glycerides of myristic and oleic acids, the former predominating, together with small quantities of resin, free myristic acid, a little volatile oil, about 0.1 per cent. of non-saponifiable oil, and a brown coloring matter. With concentrated sulphuric acid it gives a beautiful fuchsia-red color. It is used medicinally and in the manufacture of candles.

4. *Virola tallow* (*Virolafett*, G.; *suif de Virola*, F.) is obtained from *Virola sebifera*, Aublet, or *Myristica sebifera*, Swartz, the tallow nutmeg, a tree indigenous to the West Indies, Guiana, etc. The seed contains 40 to 45 per cent. of fat; it is comminuted, boiled with water, and the fat skimmed off.

At an ordinary temperature virola tallow presents a yellowish, tallow-like mass, the surface of which becomes covered with a lustrous crystalline efflorescence by age. The interior is generally brown, interspersed with small crystals. The fresh fat smells somewhat like nutmeg butter, but soon becomes rancid. It is completely soluble in ether and spirit of wine. It melts at 113° F., solidifying again at 104° F. Its specific gravity is 0.995 at 59° F. It cannot be completely saponified, consisting principally of myristin and olein and free fatty acids. In America, England, France, etc., it is used for the manufacture of soap and candles; the latter, it is claimed, being distinguished in not diffusing a disagreeable odor in burning.

*Myristica angolensis*, B., and *Myristica longifolia*, Don., are found in West Africa, and contain 60 to 70 per cent. of fat.

### *Sapoteæ* (*Styracinæ*).

1. *Shea butter, Galam butter, or Nungu Oil* (*Sheabutter*, G.; *beurre de Cé*, *beurre de Shee*, F.), is furnished by the seed of *Bassia Parkii*, D. C., or the butter tree, indigenous to northern tropical Africa. To obtain the fat the seed is comminuted

and boiled in water, or, after comminution, heated and pressed. The yield is about 40 per cent. At ordinary temperatures shea butter is of the consistency of butter and of a gray or greenish-white color, and, when fresh, of a mild and ageeable taste and odor. It keeps very long, though the fat obtained by boiling the seed in water, and brought into commerce under the names of "*nunu butter*" and "*djave fat*," becomes rancid very soon, acquiring thereby a peculiar disagreeable odor.

The specific gravity of shea butter is 0.953 to 0.955 at 59° F. It melts at between 82.5° and 84° F., and solidifies again, after having become opaque, at 73.5° F. It is very sparingly soluble in cold alcohol, but readily so in 40 parts of hot alcohol and in ether. By saponification with soda lye it yields a very hard white soap.

Shea butter consists of stearin and olein, besides many crystals of fatty acids. Palmitin is entirely wanting.

On account of the high percentage of stearin and olein, the fat is used in the manufacture of soap, and especially in that of candles, although, when fresh, it is also used as food. In medicine it is employed as an ointment in skin diseases.

We would here mention that under the names of shea butter, galam butter, and bambuck butter, was formerly understood the crude palm oil; hence the many confusions in literature.

2. *Fulwara butter*, *Ghea butter*, or *Indian butter* (*Fulwabutter*, G.; *beurre de Fulware*, F.) is obtained from the seed of *Bassia butyracea*, Roxb., or the Indian butter tree, indigenous to the Himalaya regions. The butter is prepared in the same manner as the foregoing. It is pure white, has a fine taste and odor, and is an important food and cooking fat in India. It melts at from 118.5° to 120° F., solidifying again at 95° F. Its chemical composition is similar to that of shea butter; it yields an excellent soap, and is also utilized in the manufacture of candles.

3. *Mahwa butter*, *bassia fat*, or *illipé butter* (*Mahwa butter*, G.; *huile d' illipé*, *beurre d' yllipe*, F.) is obtained from the seed of *Bassia latifolia*, Roxb., and *Bassia longifolia*, Linn.,

trees indigenous to the East Indies, especially to Malabar and Coromandel, and cultivated in most parts of India, partially for obtaining fat, and partially for the preparation of an alcoholic beverage from the flowers (*Bombay guzerate*).

In India the butter is prepared in the same simple method as the foregoing; in France, especially in Marseilles, by pressing the seed. On account of its very bitter taste the press-cake is not suitable for feeding; it contains 3 per cent. nitrogen.

When fresh mahwa butter has a mild and agreeable taste and odor, and a pale yellow or greenish-yellow color. It turns rancid in time, and becomes paler in color. It has a specific gravity of 0.972 at 59° F., melts at 109.5° to 111° F., and solidifies at 97° F. It is readily soluble in ether and partly in alcohol, and easily saponified with alkalies. It is a mixture of stearin, olein, and separated crystals of stearic acid; 20 per cent. of olein, 80 per cent. of stearin.

The seed of *Bassia longifolia* yields to petroleum-ether with a boiling point of 104° to 113° F. about 51 per cent. of fat of a yellow color. It solidifies slowly, acquiring thereby a lard-like consistency. Specific gravity 0.9175. On exposure to air and light it rapidly loses its color, and turns rancid. It is partially soluble in alcohol, completely so in ether, benzine and carbon disulphide.

| | |
|---|---|
| Fusing point of the fat | 78° F. |
| Solidifying point | 63.5–65° F. |
| Saponification number | 192.3 |

The soap prepared from the fat is hard, white and of an agreeable odor; it can fix a considerable quantity of water without losing firmness.

The fatty acids separated from the soap are white and of an agreeable odor and taste.

| | |
|---|---|
| Fusing point of the fatty acids | 103° F. |
| Solidifying point of the fatty acids | 100.4° F. |

The fatty acids are readily soluble in alcohol; they consist of

63.5 per cent. oleic acid and 36.5 per cent. palmitic acid. Like the preceding fats, mahwa butter has become an important article of commerce for England and France, though to a less degree for Germany, for the manufacture of soap and candles. In the country of its origin it is used for the same purposes, but forms also an important article for edible and culinary purposes.

Shea butter, fulwara butter and mahwa butter are brought into commerce in ordinary iron-hooped barrels of various sizes.

4. *Vitellaria paradoxa*, Gærtn., the butter-tree of the Soudan. The seed is boiled in water and the fat obtained serves various African tribes as food.

### *Laurineæ* (*Proteinæ*).

1. *Bayberry fat or expressed laurel oil* (*oleum lauri; Lorbeeröl*, G.; *huile de laurier*, F.) is obtained from the fresh or dried berries of *Laurus nobilis*, Linn., or the sweet bay, a bush or small tree indigenous to Southern Europe. The fresh and ripe berries are bruised in a mortar, boiled afterwards for three hours in water, and then pressed in a sack. The expressed oil is then mixed with the decoction, and when cold the butyraceous oil is found floating on the surface. From the dried berries it is procured by exposing them to the vapor of water until they are thoroughly soaked and then rapidly subjecting them to pressure between heated metallic plates.

Bayberry fat is a mixture of volatile oil, laurostearin, myristin and resin. It has a butyraceous consistency and a granular appearance. Its color is greenish, its odor that of the berries, and its taste bitter and aromatic. It melts at 86° to 95° F. It is completely soluble in ether, while alcohol dissolves only the volatile oil and the green coloring matter.

Its behavior towards ether and alcohol serves as an excellent test. Bayberry fat adulterated with lard does not give with ether a clear, but a milky, turbid solution. Artificially colored mixtures of fat act in the same manner and are not granular.

Bayberry fat is occasionally employed externally as a stimulating liniment in sprains and bruises, as well as in paralysis. Its principal use, however, is in veterinary medicine.

2. *Laurel tallow or undung* (*Lorbeertalg*, G.), a solid fat is obtained by boiling the seed of *Tetranthera laurifolia*, Jacq., a tree of Cochin China and Java. It is used in the manufacture of candles.

3. *Tangkallah fat or Minjak tangkallah* (Java) (*Tangkallahfett*, G., *beurre de Tangkallah*, F.), is furnished by the seed of *Cylicodaphne sebifera*, Bl., of West Java. It melts at 113° F., consists of 14 per cent. of olein and 85 per cent. of laurostearin, and is used in the manufacture of soap and candles. The fruit of one tree suffices for the manufacture of five hundred candles.

4. *Alligator pear oil or avocado oil* (*Advogatofett*, G., *huile d', avocatier*, F.), from *Persea gratissima*, Gaert., a tree indigenous to North Brazil and the West Indies, and transplanted into the East Indies. The oil is of a slightly dark greenish-brown color, and consists of 30 parts of olein and 70 parts of laurostearin and palmitin; it is used in the manufacture of soap, especially in America. Treated with alkalies it very much resembles palm oil.

### *Palmæ* (*Spadicifloræ*).

1. *Palm oil or palm butter* (*oleum palmæ; Palmöl*, G.; *beurre de palme*, F.) is obtained from the fruit of several species of palms, principally of *Elæis guineensis*, Jacq , or the African oil palm, and *Elæis melanococca*, Gaert. Both varieties are indigenous to Africa, and are also cultivated in South America. The nut or fruit is yellow, brown, or red, and has a somewhat hard pulp rendered fibrous by vascular bundles, in the cells of which the oil is found in the form of drops or lumps.

From this pulp, which smells like orris root, the oil, if it is to be used for table purposes, is obtained by pressing or boiling with water. In the manufacture of the ordinary commercial article less care, however, is exercised. To save boiling in water the fruit is allowed to rot in piles, then pounded with pestles, and the hard kernel removed. The pulp is then pressed in bags made from the fibres of palm leaves. Sometimes the residue in the bags is boiled with water and the oil collecting on the surface skimmed off. As the product obtained by this primitive manner of manufacture is mostly impure and frequently intentionally contaminated, it is generally purified on board the vessels by boiling with water in large boilers.

Fresh palm oil has a dark yellow or red yellow color, an odor resembling that of orris root, and a sweetish taste. On exposure to the air it very soon becomes rancid, and pale in color. At a medium temperature it is of the consistency of butter or lard. Its fusing point ranges from 80.5° to 97° F.; its solidifying point from 70° to 80.5° F. With the increase of the formation of free acids the fusing point of old oils rises to 108.5° F. Palm oil, consisting of palmitin and olein, is, even when fresh, always more or less in that condition in which other oils are called rancid because it contains considerable quantities of fatty acids in a free state, instead of being fixed on glycerin. In fresh palm oil, for instance, is found one third, and in palm oil, which fuses at 104° F., four-fifths of its weight in free fatty acids, which can be separated from the olein, glycerin, and undecomposed palmitin by pressure. The glycerin can also be extracted with water. It is remarkable that the quantity of glycerin separated by the oil becoming rancid decreases in very old oil.

The cause of the rapid and progressive decomposition of palm oil, which is out of all proportion to that of ordinary oils, will, no doubt, be found in the admixture of various constituents of the palm fruit which readily undergo a change and act as ferments.

Palm oil is readily and completely soluble in ether. In cold alcohol it dissolves with great difficulty, but with somewhat greater ease in boiling alcohol, though, on cooling, the excess is again separated. Its specific gravity is 0.945 at 59° F. It is readily saponified with alkalies, yielding a yellow soap. The yellow-red coloring matter is not destroyed by saponification, but is readily removed by the action of light, air, heat and oxidizing agents, such as chlorine, oxygen, or strong acids. The coloring substance cannot be extracted with water or spirit of wine, as it is not suspended in the fat, as in nutmeg butter or bayberry fat, but dissolved.

In consequence of a certain content of water adhering to it, palm-oil commences to bubble at 239° F., the ebullition continuing to about 374° F. At 284° F. white acid vapors of an acrid odor are formed, which become very annoying at 374° F., though they do not originate from acrolein. They amount to very little as regards weight, and do not produce an ebullition of the oil at 392° F., or even at 464° F. Palm oil thus heated shows after cooling no trace of the yellow-red color; it is white with a brownish tint, which is due to the vegetable remains in the oil decomposed by heat. The destruction of the yellow-red color does not depend on the co-operation of light and air; it is accomplished in closed as well as in open vessels.

At 580° F. the oil commences to boil under decomposition, an odor of acrolein is developed, and fatty acids pass over.

To bleach palm-oil at a lower temperature by the agency of air and light, it is heated to 212° F., and at this temperature exposed to the air, which is accomplished by various mechanical appliances. The most simple is to fill a fine-meshed sieve fastened to a long wooden handle, with oil, raise it up quickly, and bring the oil in contact with the atmospheric air by allowing it to run back into the boiler. By frequently repeating this rather slow process, the bleaching of the oil is accomplished, though, as a rule, it is incomplete. A better arrangement is a kind of water-wheel so adjusted that one-half of it dips into the oil. By setting the wheel in motion the paddles lift up a portion of the oil, allowing it to run back in a fine stream into the boiler.

Bleaching palm oil by air and light is now seldom practised. The chemical means of bleaching are generally mentioned in the chapter on "Bleaching Fats."

Palm oil is used in the manufacture of soap, candles, and stearin, even the oldest and most rancid oil being adapted for these purposes.

The principal places in Africa from which palm oil is exported are: The territory of the Bennin river, Cape Palmas and neighborhood, Lagos—the most highly esteemed varieties are prime and secunda Lagos, the first being more readily bleached than the latter—Porto Novo and neighborhood, Whydah, etc. As far back as 1836, England received 13,850 tons, in 1861 over 37,000 tons, and at present more than 50,000 tons are annually exported from the African coast.

2. *Palm-nut*, or *palm-kernel oil* (*Palm-kernöl*, G.; *huile de pepin de palme*, F.) is contained in the stony kernels of the fruit of the above-mentioned palms. The kernels were formerly thrown away or burnt, though the negroes were well acquainted with the fact that they contained oil. As the preparation of the oil is connected with difficulties, it is scarcely ever attempted in the localities where the palms grow, except in the Mombattee regions, where the oil is extracted by placing the kernels in stone jugs, which are buried in the ground, and a fire built over them. The oil thus obtained has a disagreeable empyreumatic odor.

The manufacture of palm-kernel oil is now carried on in many places according to the most improved methods. The first kernels were brought to England in the commencement of the fifties, and later on shipments were made to Marseilles where the industry has assumed great proportions. Only the clean kernels freed from the hard woody shell are shipped to Europe.

The manufacture of palm-kernel oil in Europe is chiefly concentrated in Marseilles, France and Harburg, Germany; there are also factories in Liverpool, London, Cologne, Gross-Gerau, Obertürkheim near Stuttgart, Magdeburg, Berlin and Nantes. Of late years the importation of palm-kernel has increased to an enormous extent; for instance, in 1872, Marseilles imported about 13,000 tons; in 1880, about 35,000 tons, while at present thehimportation amounts to an average of 40,000 tons.

Harburg at present uses in its four factories 35,000 tons of kernels per annum, while Europe altogether consumes over 100,000 tons.

The content of oil in the kernels varies very much—from 44 to 54 per cent.; though sometimes they contain as much as 60 per cent., and even 62 per cent. Kernels from New Granada and Brazil are much smaller than the African, and contain scarcely 40 per cent. of oil.

| Kernels from: | Sherbro. | Quittha. | Old Calabar. |
|---|---|---|---|
| Fat | 45.40 p. c. | 46.85 p. c. | 53.80 p. c. |
| Organic substances. | 43.35 " | 43.20 " | 36.40 " |
| nitrogenous substances therein | 8.60 p. c. | 7.90 p. c. | 8.20 p.c. |
| Ash | 1.80 " | 1.55 " | 1.65 " |
| Water | 9.45 " | 8.40 " | 8.15 " |
| | 100.00 " | 100.00 " | 100.00 " |

| | Palm-kernel cake. | | Palm-kernel meal freed from oil. | |
|---|---|---|---|---|
| Fat | 9.10 p. c. | | 1.86 p. c. | |
| Organic substances | 79.84 " | | 84.20 " | |
| protein substances therein. | | 16.66 p. c. | | 16.70 p. c. |
| Ash | 3.90 " | | 4.12 " | |
| Water | 7.16 " | | 9.82 " | |
| | 100.00 " | | 100.00 " | |

The ash of palm-kernel cake contains:

| | |
|---|---|
| Potash | 19.10 per cent. |
| Soda | 0.88 " |
| Magnesia | 17.19 " |
| Lime | 11.96 " |
| Ferric oxide, etc | 3.54 " |
| Chlorine | 0.22 " |
| Sulphuric acid | 2.02 " |
| Silica | 2.94 " |
| PHOSPHORIC ACID | 42.15 " |
| | 100.00 " |

In working the kernels, comminution and preparation for pressing are of the utmost importance. The finer the material is brought into the press, the better the yield will be, and the easier it will be obtained. The large percentage of fat and tenacious vegetable albumen in the kernels renders comminution extremely difficult, so that the material can only be moderately reduced by means of rolls, or a mill, necessitating a repetition of the process for a more complete comminution. For the latter process French mills or smooth rolls are very well adapted.

Presses with plates which can be heated are recommended for pressing.

The kernels are frequently extracted by means of naphtha or carbon disulphide. The oil obtained by expression has a deeper yellow color than that by extraction.

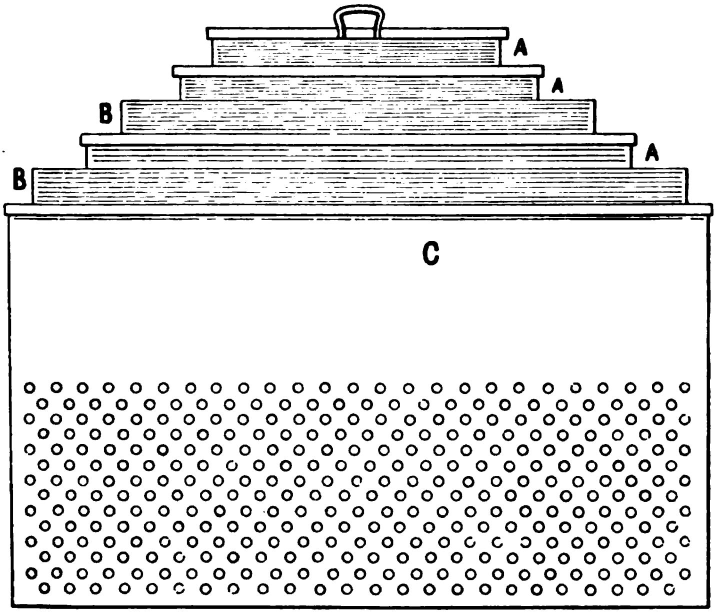
As the pressed oil contains vegetable mucus and albumen, it must be allowed to rest for a long time in a melted state for purification and clarification. Purification by sulphuric acid being tedious, Schneider's apparatus, shown in Figs. 232 and 233, is well adapted for the purpose.

The apparatus is very compact, but can be easily taken apart and cleansed. It contains three series of boxes made of wood and sheet iron, viz., the decanting boxes *A* and the filtering boxes *B*, which sit in the sieve-box *C*. All the boxes are open on the top. The boxes *A* consist each of a bottom and four vertical sides, each provided with a horizontal slit *D* near the upper edge. The jacket *E* surrounds the boxes and joins their sides above the slit *D*. The filtering boxes *B* consist of bottoms and framework sides, the latter being covered with linen or some other filtering material. The lower halves of the sides of the sieve-box *C* are perforated.

The filtering boxes *B* and the sieve-box *C* are provided with valves *F*, which are set by the valve-rod *G*, the sieve-box being besides provided with a discharge-cock *H*.

The apparatus works as follows: The sieve-box *C* is suspended over the reservoir which is to receive the clarified oil, and the decanting boxes *A* and filtering boxes *B* are placed upon the supports *J* in the sieve-box *C*. The oil to be clarified is introduced through the pipe *K* into the innermost decanting box *A*, passes through the slit *D* into the second box *A*, and from here into the filtering box *B*, and after penetrating through the filtering material with which the sides are covered, arrives finally in the sieve-box *C*, and leaves it through the perforations.

FIG. 232.

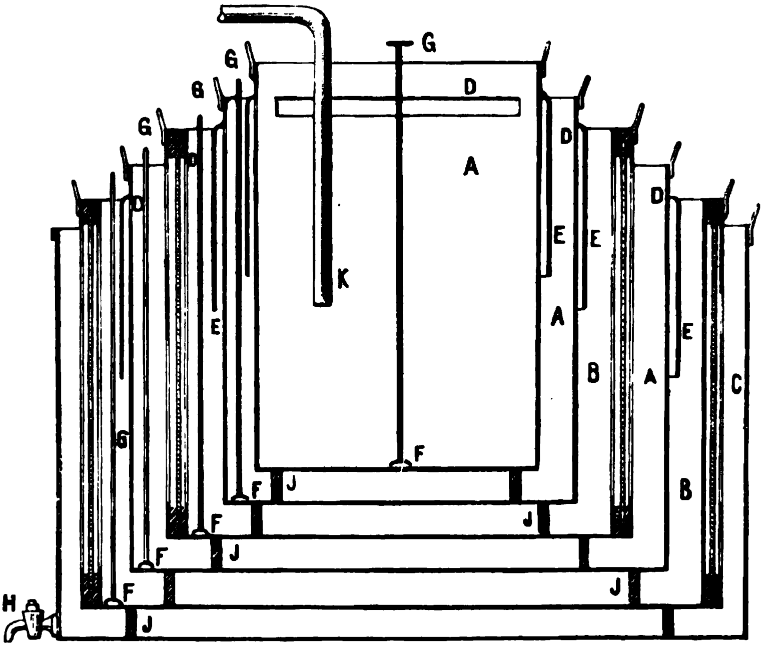


*A*, decanting boxes; *B*, filtering boxes; *C*, sieve-box.

The impurities are precipitated on the bottoms of the boxes and the filtering material, which, of course, must be occasionally cleansed. This is effected by means of the valves *F* and the cock *H*.

As previously mentioned, palm-kernel oil obtained by extraction is white, and that by expression yellow or gray. The fat is granular, and when fresh has a mild and agreeable taste and odor; it becomes, however, very soon rancid and acquires an acrid odor, resembling that of cocoanut oil. It has a specific gravity of 0.952 at 59° F., fuses at 77° to 79° F., and solidifies at 69° F. Old oil melts at 80.5° to 82.5° F. It is scarcely soluble in cold alcohol, somewhat more so in hot alcohol, and gives a clear solution with 3 parts of ether. It consists of palmitin and olein, and when freshly extracted contains no free fatty acids, which, however, are found in the old oil.

FIG. 233.



*D*, slit in *A*; *E*, jacket around *A*; *F*, discharge-valves; *G*, valve-rods; *H*, discharge-cock; *J*, supports; *K*, feed-pipe.

The fatty acid mixture separated after saponification of the oil by acids consisis of caproic, caprylic, capric, lauric, myristic, palmitic and oleic acids in quite varying relative proportions. As in cocoanut oil, lauric acid forms the principal constituent.

| | |
|---|---|
| Saponification number of the oil | 258–265 |
| Iodine number | 10.3–17.5 |
| Fusing point of the fatty acids | 77°–83° F. |

Palm-kernel oil is chiefly used in the manufacture of soap and candles; to what extent may be judged from the quantity of kernels imported as mentioned above, which represents about 40,000 tons of oil.

3. *Tucum oil* (*Aouaraöl,* G.; *huile d'Aouara,* F.) is obtained from the fruit of *Astrocaryum vulgare,* Mart., a species of palm indigenous to Guiana. It is manufactured in a similar manner to palm oil. It has a cinnabar-red color, the coloring substance being dissolved in the oil itself like that of palm oil. Tucum oil is a semi-fluid fat of an agreeable taste and odor, does not become rancid as easily as palm oil, and contains more olein. It is used in the manufacture of soap.

4. *Cocoanut oil* (*oleum cocois; Cocosöl,* G.; *huile de Coco, beurre de Coco,* F.) is obtained from the seed of *Cocos nucifera,* Linn., or the genuine cocoa palm, indigenous to the islands of the South Sea and the East Indian archipelago, but now cultivated in many tropical countries, especially in Java and the East Indies. The pulp of the cocoanut is called "copra" or "copperah," and hence the oil is sometimes called copra oil. Copra contains 60 to 70 per cent. of fat.

| | Indian copra. | African copra. |
|---|---|---|
| Oil | 68.75 p. c. | 66.80 p. c. |
| Organic substances | 23.65 " | 25.25 " |
| albuminous substances therein | 9.16 p. c. | 10.20 p. c. |
| Ash | 1.45 " | 1.50 " |
| Water | 6.15 " | 6.45 " |
| | 100.00 " | 100.00 " |

| | Cocoanut cake. | Cocoanut meal freed from oil. |
|---|---|---|
| Fat | 10.75 p. c. | 1.85 p. c. |
| Organic substances | 77.20 " | 87.65 " |
| nitrogenous substances therein | 21.90 p. c. | 23.20 p. c. |
| Ash | 6.25 " | 5.95 " |
| Water | 5.80 " | 4.55 " |
| | 100.00 " | 100.00 " |

The ash contains:

| | | |
|---|---|---|
| Potash | 40.23 | per cent. |
| Soda | 3.14 | " |
| Magnesia | 2.80 | " |
| Lime | 5.00 | " |
| Iron | 3.60 | " |
| Chlorine | 14.32 | " |
| Sulphuric acid | 2.50 | " |
| Silica | 4.11 | " |
| PHOSPHORIC ACID | 24.30 | " |
| | 100.00 | " |

To prepare the oil the copra is freed from the shells, boiled in water for some time, then bruised in mortars and pressed. The resulting milky mass is heated in large boilers and the oil collecting on the surface skimmed off. The residue, which is called "*poonak*," is an excellent cattle feed.

In Malabar the copra is comminuted, dried upon frames over a coal fire, then upon mats in the sun, and finally pressed.

In Tahiti the natives are too lazy to undertake the labor of pressing. They simply bruise the copra, and expose the bruised mass in perforated pots to the sun, the oil running off into vessels under the pots.

The most rational arrangements for the manufacture of this important article of commerce are found in the oil mills of Ceylon, conducted by Europeans. Here the copra is ground, the ground flour is packed in hydraulic presses and subjected to two pressings, the last of 6000 pounds to the square inch. Recently the dried copra itself is brought to Europe, where it is expressed in the same manner as palm-kernels.

The copra contains two fats of varying consistency, which, in pressing, can be separated or mixed together. In consequence of these properties, either a liquid, soft, or solid fat can be obtained, according to the force of pressure and the temperature at which expression is effected.

Fresh cocoanut oil has a beautiful white color, a peculiar mild taste, and an agreeable odor, but it has a great tendency to become rancid, acquiring thereby a peculiar sharp odor and acrid taste. Fresh cocoanut oil fuses at 72.5° F., and the ordinary commercial article at 75° to 76° F., and solidifies slowly at 68° to 69° F. The cold-drawn oil, which is used as table oil by the natives and seldom brought into commerce, is greenish white, commences to fuse at below 68° F., and solidifies again at 53.5° to 55.5° F. Brazilian cocoanut oil has generally a higher fusing point, 79.0° to 80.5° F.

By heating cocoanut oil slowly to from 320° to 338° F. an acrid odor, resembling that of lactic acid, is developed, and the oil on cooling solidifies with great difficulty, and not to a white mass, but watery. By heating for some time to 464° F. the oil acquires the property of remaining fluid for days after cooling. By heating to from 536° to 572° F. acrolein is developed.

As regards the solubility of cocoanut oil in alcohol, it stands intermediate between palm oil and palm-kernel oil and shea butter, *i. e.*, it is more soluble than palm oil and less so than shea butter; in ether, volatile and fat oils it is readily soluble.

In saponifying, cocoanut oil exhibits a peculiar behavior as compared with other oils and fats. It cannot be saponified with dilute lyes even when heated; the oil floats upon the boiling lye without being attacked by it. Heated strong lyes saponify the fat, but saponification takes place very suddenly and the formed soap is so hard that it can scarcely be cut.

In the cold, and at a medium temperature, cocoanut oil is readily saponified, but the formed soap shows the peculiarity of being soluble in salt water and not solidifying therein on cooling. The solidifying soap formed by cold saponification can absorb a considerable quantity of water, while the formed glycerin and lye, etc., nevertheless solidify quickly to a hard mass and give a quick and plentiful foam with water. For this reason cocoanut oil is used for cosmetic soaps and cheap white "filled" soaps, especially as it transfers the property of saponifying in the cold to a mixture with other fats, such as tallow, bone fat, etc.

Cocoanut oil contains the glycerides of volatile fatty acids, caproic, capric, and especially caprylic acids; of the non-volatile fatty acids, lauric, myristic and palmitic acids. Stearic acid is not present, or at least in extremely small quantities.

By long heating cocoanut oil with nitric acid, suberic, pimelic, lepargylic, adipic, succinic and nitro-capric acids are formed.

Besides in the manufacture of soap, the more solid fatty acids of cocoanut oil, which are separated by pressing from those more readily melted, are used in the manufacture of candles.

Cocoanut oil is chiefly manufactured on the coast of Malabar and in Ceylon; it is brought into commerce from Cochin China and Colombo.

Cochin superior and Ceylon prima are considered the best qualities of cocoanut oil.

5, *Macaja butter* (*Macaja butter*, G., *huile de Macaya*, F.), is obtained from the fruit of *Acrocomia sclerocarpus*, Mart., or the Macasuba palm, which is indigenous to the West Indies, Brazil and Guiana. The fat, of which the fruit contains 60 to 70 per cent., is yellowish, smells like violets, and is largely used in the manufacture of toilet soaps in Brazil and the West Indies. It has been recently brought into European commerce.

6. *Cohune oil* (*Cohuneöl*, G.; *huile de Cohune*, F.), from *Attalea Cohune*, Mart., or the Cohune palm. Large forests of this palm are found in the British Honduras. The fruit yields a sort of cocoanut oil, but inferior to it.

7. *Comou butter* (*Comuöl*, G.; *huile de Comou*, F.), is expressed from the fruit of *Oenocarpus bacaba*, indigenous to Central and South America, and is used in the manufacture of soap and candles.

8. *Muriti fat* (*Muritifett*, G.; *huile de Muriti*, F.), from *Mauritia vinifera*, Mart., the wine-palm or maurice palm, indigenous to Brazil, especially Orinoco.

9. *Tourlourou oil* (*Turlurufett*, G.; *huile de Tourlourou*, F.), is obtained from the fruit of *Manicaria saccifera*, Gaert., indigenous to the forests of Brazil, Guiana, and the West Indies, and is used in the manufacture of soap.

We would here remark that under this name is also known an oil which is prepared from the intestines of *Cancer ruricola*, Linn., or the Turluru crab. The animal, which is of a reddish to blood-red color, visits the shore during the rainy season, and is often caught by the millions. It is an ordinary sea-crab, found in all the southern seas. On the Senegal the negroes use the oil as an embrocation against rheumatism, and also for rubbing with it the entire body to protect it, on the one hand, from the sun, and, on the other, from moisture. The oil is also used in France.

10. *Para butter* (*Parapalmöl*, G.; *huile d' Assay*, F.), from the fruit of *Euterpe oleracea*, Mart., or the common cabbage palm, indigenous to Brazil.

## B. SOLID FATS OF THE ANIMAL KINGDOM.

### *Multungula, Pachydermata* (*Setigera*).

*Lard* (*adeps, adeps suillus; Schweinefett*, G.; *graisse de porc, axonge*, F.) is obtained from *Sus scrofa domesticus*, Linn., or the hog. The fat of the hog varies according to the part of the body from which it is taken. The exterior envelope of fat, which surrounds the entire body, especially the back and sides, immediately beneath the skin, melts much more readily than the fat deposited along the ribs, intestines, and kidneys. The best season in the year for collecting the fat and preparing lard is in January and February, as at that time the fat is of greater consistency than during the hot months. In consequence of the increased respiration in the denser atmosphere during the cold season, the deposited fat evidently contains less hydrogen and more carbon, as the hydrogen, having at this temperature a greater affinity for oxygen, combines in the first place with it during the act of respiration. Summer fat is therefore richer in olein and winter fat in stearin. Feeding and the sanitary condition of the pig exert also considerable influence upon the consistency of the fat; hence soft and smeary fat is also frequently found in the cold season. Pigs exclusively fattened on acorns always yield a somewhat yellowish lard. The leaf fat of young pigs gives the best lard, as regards appearance and consistency, and the largest yield.

The manner of rendering lard has already been described in Vol. 1, p. 395, and in addition to the different varieties of lard mentioned there, a description of some other hog-fat products may prove useful.

*White grease.*[*] This grease is made chiefly from hogs which die in transit, by being smothered or frozen. Formerly it was also made from animals dead of disease; but this product has of late been diminished on account of certain State laws requiring the carcases of hogs which have died of cholera to be buried. This grease is made from the whole animal with the exception of the intestines. The latter are rendered separately and make "brown grease." The rendering is done in closed tanks at a high pressure. The residue is used in the manufacture of fertilizers. White and brown grease are chiefly used in the manufacture of low grade lard oils and soap.

*Yellow grease.* Yellow grease is made by packers. All the refuse materials of the packing houses go into the yellow grease tank, together with any hogs which may die on the packers' hands. Yellow grease is intermediate in value between white and brown. It is used for the same purpose.

*Pigs'-foot grease.* This grease is obtained chiefly from the glue factories, and is used for making lard oils and soap.

Pure lard is white, granular, of the consistency of salve, has a slight, peculiar odor and an agreeable, sweetish, oily taste. Exposed to the air and light, it becomes yellowish to yellow and acquires a rancid, acrid taste. It liquefies between the fingers, its melting point lying between 89.5° to 91.5° F. It solidifies at 78.8° F., whereby a development of heat to 86° F. takes place. After cooling it acquires an incomplete crystalline structure. Its specific gravity is 0.938 to 0.940 at 59° F. It is soluble in 27 parts of absolute alcohol and in 35 parts of alcohol of 0.9128 specific gravity, in ether, chloroform, carbon disulphide, hot benzine, and heated petroleum-spirit. Fresh lard shows no acid reaction, and by saponification and subsequent decomposition of the soap yields 8.80 per cent. of glycerin, and 95.90 per cent. of stearic, palmitic, and oleic acids, 4.70 per cent. of the 104.70 per cent. being water absorbed for the formation of glycerin and acid.

---

[*] U. S. Department of Agl., Div. of Chem. Bull. No. 13. Lard and Lard Adulterants, by H. W. Wiley, 1889.

Lard is composed of:—

| | | |
|---|---|---|
| Carbon | 76.54 | per cent. |
| Hydrogen | 11.94 | " |
| Oxygen | 11.52 | " |
| | 100.00 | " |

Subjected to great pressure at 32° F. it consists of 60 parts of a colorless oil which does not freeze on exposure to severe cold, and 38 parts of margarin, palmitin and stearin. This property is utilized for the manufacture of

*Lard oil* (*Schmalzöl*, *Specköl*, G.; *huile de graisse*, F.) The lard is wrapped in cakes with cloth, each cake containing 10 to 20 pounds. The cakes are then placed in a large press with suitable partitions to facilitate the egress of the oil. These presses are sometimes 40 to 50 feet in length, and when first filled 12 to 18 feet high. The pressure is applied very gradually at first by means of a lever working a capstan, about which the chain is wrapped, attached to the upper part of the press.

Lard oil is limpid like olive oil, but of a pale yellow color. As, according to the manner of preparation, it contains varying quantities of palmitin and stearin, its specific gravity and freezing point vary very much. The average specific gravity of the commercial article is 0.9165 at 59° F., and the freezing point 30.0 to 23.0° F. It is used for burning and lubricating purposes, and largely for greasing wool, for which it is said to be superior to olive oil, and preferable from its low price. In France it is also employed in the adulteration of olive oil. Large quantities of lard oil are made in the United States, the value of the output of seven factories, reported by the Extra Census Bulletin of March 15, 1894, being $1,181,613.

The solid residue remaining in the presses forms what is known as *lard stearin*. The lard stearin prepared from prime steam lard is used for making compound lard. It has about 0.5 per cent. free fatty acid (less than lard oil), and crystallizes in long needles, making the texture tough. It also forms an excellent material for the preparation of the better qualities of soap and the manufacture of candles. It is brought into commerce principally from Cincinnati under the name of "solar stearin," and is largely exported to England, France and Germany.

The most common adulteration of lard is with *water*, to increase the weight, which is readily detected by careful melting of a sample, and allowing it to stand in a lukewarm place, whereby the water is separated on the bottom of the test-tube.

To increase the weight, dilute *solutions of soda*, *potash*, and *soda lye* are also used, which at the same time give the lard a whiter appearance; further, *chalk*, *alum* and *clay*. To detect such adulterations melt about 100 parts by weight of the suspected lard with an equal or 1½ times the quantity of distilled water at 140° to 158° F., and allow it to rest for a few hours.

*Chalk* or *clay*, if present, will settle on the bottom; *common salt* is detected in the aqueous fluid by the white precipitate formed with solution of nitrate of silver; *alum* by the voluminous precipitate with ammonia, and the white precipitate by the addition of barium chloride; *soda* and *potash solution*, *oils*, and *lye*, by the precipitate with calcium chloride, the latter specially by the yellow color produced by mercuric chloride.

Admixtures of *starch* and *flour* give, when treated with iodine solution, iodine starch. Occasionally it happens that the lard contains traces of *copper* or *lead* from the copper or glazed vessels used in rendering. To determine these the lard is digested with dilute acetic acid and a portion of the clear extract compounded with sulphide of hydrogen, which produces a browning or blackening. For the special detection of *copper* another portion of the clear extract is compounded with solution of potassium ferrocyanide, which gives a brown precipitate; while for the detection of *lead* dilute sulphuric acid is added to the third portion of the extract, which produces a white precipitate.

For the establishment of adulterations with other fats lard has to be subjected to a very thorough examination.

The principal materials used for sophisticating lard are refined cotton-seed oil and pure pressed beef fat—*oleo-stearin.*

The term *refined* lard has long been used to designate a lard composed chiefly of cotton-seed oil and lard-stearin. The largest manufacturers of this kind of lard have now abandoned this term and are using the label " lard compound " instead. The prime steam lard in a state of fusion, the stearin also in a liquid condition, and the refined cotton-seed oil, are measured in the proportions to be used and placed in a tank at a temperature of 120° to 160° F. In this tank the ingredients are thoroughly mixed by means of paddles operated by machinery. After mixing, the compound lard passes at once to artificial coolers, where it is chilled as soon as possible. It is thence run directly into small tin cans or large packages and prepared for market.

Since the addition of cotton-seed oil renders the lard too soft, this disadvantage is frequently overcome by the use of *oleo-stearin* from beef suet. This product is chiefly made from the caul fat of beeves. This fat is rendered in open kettles at a low temperature. The resulting tallow is placed in cars in a granulating room, where it is allowed to remain for thirty-six to forty-eight hours at a temperature of 80° to 90° F. The contents of the cars are then mixed and placed on a revolving table, where they are made into cakes. These are wrapped with stout cotton cloth and placed in a powerful press, where a gradual pressure at 90° F., becoming very strong at the end, is applied for one or two hours. The expressed oil, known as *oleo-oil*, is used in the manufacture of butterine. The stearine is removed from the press as white hard cakes, and is used for adulterating lard. The oil is sometimes filtered with a small percentage of fuller's earth, to improve its color and brightness.

Besides purified cotton-seed oil and oleo-stearin, other fats and oils are also occasionally used for the adulteration of lard. For the detection of all such additions simple reactions have been repeatedly proposed, viz:

*Alcoholic solution of nitrate of silver* (Vol. 1, p. 177). Pure lard will stand this test, but only such as has been prepared on a small scale with all possible precautions; most commercial varieties, however, will not stand it. Nevertheless one would not be justified in calling them adulterated because extremely small quantities of foreign substances, which almost invariably occur in commercial lard, suffice to brown silver solution.

The same applies to *solution of phospho-molybdic acid.* This test also is extremely sensitive, so that most commercial varieties will not stand it. The test is carried out as follows:

To 1 gramme of the fat in question, previously dissolved in 5 cubic centimeters chloroform, add 2 or 3 c.c. of solution of sodium phospho-molybdate and shake vigorously. After standing quietly for a few minutes the two layers of fluid separate, and the supernatant solution of the reagent appears colored more or less intensely green to green-blue according to content of reducing substances in the lard. By means of the so-called *Hirschsohn's reaction* it is claimed an addition of 5 per cent. of cotton-seed oil to lard may be recognized. To 5 grammes of the fat to be tested add 5 to 6 drops of a solution of 1 gramme of chloride of gold in 200 grammes of chloroform, shake, and immerse in boiling water for 20 minutes. In the presence of cotton-seed oil the mixture with the light falling upon it shows a rose to red color, and with transmitted light a greenish color.

*Labiche's test.* Add to the melted fat basic acetate of lead solution and, after shaking, ammonia. A more or less orange color is claimed to indicate cotton-seed oil. This test is of no more value than the preceding.

Hence, as previously mentioned, reliable results can be obtained only by a thorough examination comprising the microscopic picture of the crystallized fat, determination of the fusing point of the fat, of the fusing and solidifying points of the fatty acids, of the saponification number and of the iodine number.

The microscopic examination of lard for the detection of tallow is readily executed, according to Labler, by testing the crystals separated from solution in ether or benzin. Crystals of pure lard form feathery masses radiating from a longitudinal axis with similar secondary branches. The beef-fat crystals, on the other hand, form spherical masses radiating from a common center, breaking up under the cover-glass into fan-shaped clusters, often with a peculiar twisted appearance.

The specific gravity of lard containing cotton-seed oil is higher than that of the pure fat, viz., 0.862 to 0.864 at 212° F. The same difference is also shown when the fluid portion of the oil is isolated by expressing 2.2 pounds of lard in linen and filtering the fluid. Compound lard gives 30 to 40 per cent. of oil of specific gravity 0.916 to 0.918 at 64.4° F., while the specific gravity of pure lard is 0.912 to 0.914.

| | |
|---|---|
| Saponification number of lard | 195–196 |
| Fusing point of the fatty acids | 100.4°–102.2° F. |
| Solidifying point of the fatty acids | 95° F. |
| Iodine number of lard | 59–60. |

The iodine number of compound lard is, as a rule, much higher than that of pure lard, but mixtures showing the correct iodine number can also be prepared.

According to Langfurth better results are obtained by calculating the iodine number to oleic acid and comparing it with the quantity of oleic acid found from the solidifying point. With adulterated lard, the former is always greater than the latter, with pure lard the same or smaller.

Perfectly reliable results are obtained by determining, according to Muter and Koningh, the iodine number of the fluid portion of the fatty acids which, with pure lard is 94, with tallow 90, and with cotton seed oil 136. If the iodine number is above 94, the sample is adulterated.

According to Asboth, the content of cotton-seed oil may be approximately calculated from the iodine number of the fatty acids: Cotton-seed oil contains 70 per cent. fluid acids, lard 54.3 per cent. If, according to Muter and Koningh, the iodine number has been found $= i$, the content of cotton-seed oil acid in the fluid fatty acids is

$$x : 100 = i - 94 : 136 - 94$$

and

$$x = \frac{100\,i -}{42}.$$

Since the fat further contains $a$ per cent. and pure cotton-seed oil 70 per cent. fluid fatty acids, the content of cotton-seed oil $C$ in the fat is

$$C = \frac{a}{70}\,x = \frac{10\,a\,(i-94)}{294}.$$

According to this formula the content of cotton-seed oil can also be calculated when, besides lard, other fats of animal origin, such as oleo-stearin, tallow, etc., are present.

In the United States the great seat of the lard trade is in the Mississipi Valley, which takes in the States of Illinois, Missouri, Ohio, Indiana, Iowa, Kentucky, and a few others. In Cincinnati, Chicago, and other cities, are found enormous establishments, with the most perfect arrangements for rendering lard. In the investigations, some years ago, before the Committee on Agriculture of the Senate and House of Representatives, it was developed that the annual production of lard in the United States was then 600,000,000 pounds, of which about half was pure lard and the other half pure lard mixed with stearine and cotton oil, the "refined" or compound lard of commerce. The annual exports of lard were then about 320,000,000 pounds, of which about 40 per cent. were compound or refined lard.* These figures are now greatly increased.

If we take the percentage of cotton oil in the above quantity compound lard of at 40, the total weight of oil used in manufacturing mixed lard is 120,000,000 pounds.

According to the figures furnished by the Bureau of Statistics, the production of lard from 1877 to 1887, inclusive, was as follows:

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* Statement of Mr. G. H. Webster before House Committee on Agriculture. Report of Hearings, p. 26.

| Years. | Total, Pounds. | Years. | Total, Pounds. |
|---|---|---|---|
| 1886–'87 .......... | 527,032,000 | 1881–'82 .......... | 468,929,000 |
| 1885–'86 .......... | 514,230,000 | 1880–'81 .......... | 517,660,000 |
| 1884–'85 .......... | 480,405,000 | 1879–'80 .......... | 479,020,000 |
| 1883–'84 .......... | 444,450,000 | 1878–'79 .......... | 514,295,000 |
| 1882–'83 .......... | 419,513,000 | 1877–'78 .......... | 404,572,000 |

The exports for the fiscal years ending June 30 1885 to 1895 were as follows:

| Years. | Lard exported. Pounds. | Years. | Lard exported. Pounds. |
|---|---|---|---|
| 1885.............. | 283,216,339 | 1891.............. | 498,343,927 |
| 1886.............. | 293,728,019 | 1892.............. | 460,045,776 |
| 1887.............. | 321,533,746 | 1893.............. | 365,693,501 |
| 1888.............. | 297,740,207 | 1894.............. | 447,566,867 |
| 1889.............. | 318,242,990 | 1895.............. | 474,895,274 |
| 1890.............. | 471,083,598 | | |

The cost of materials used by 17 lard-refining establishments reported by the Extra Census Bulletin of March 15, 1894, amounts to $12,654,320, and the value of their products to $15,474,848.

Considerable quantities of cotton-seed oil are also used in the manufacture of *cottolene*, which is brought into commerce as a substitute for lard. It contains no hog's grease whatever. According to C. Bischoff, it is a mixture of cotton-seed oil with oleo-stearin in the proportion of 75 per cent. of the former and 25 per cent. of the latter.

The principal use of lard is for culinary purposes and in pharmacy, where it is employed as a basis for ointments, pomades, etc.

### *Solidungula.*

*Horse fat* (*Kammfett*, G.; *huile de la crinière*, F.), is obtained from the upper portion of the neck of *Equus caballus*, Linn., or the horse. Now that the butchering of horses is carried on on a large scale, the fat of the other portions of the body is also rendered out and brought into market. The genuine horse fat from the upper portion of the neck is pure white, odorless, of less consistency than lard, fuses at 89.5° F., and consists of 70 parts of olein and 30 parts of a mixture of stearin and palmitin. Other horse fat is thickly fluid, and, when at rest, separates into a solid and an oily part, acquiring the consistency of lard only at 50° F., and of butter at 46.4° F. It has a somewhat dirty, slightly yellowish-brown color, and a peculiar fatty odor.

According to Filsinger, horse fat has a specific gravity of 0.9189 at 59° F. Its iodine number is 84.0, saponification number, 197.1, and acid number, 1.7. It is frequently adulterated with cheaper bone oils.

Horse fat is used for ointments by veterinary surgeons, but is principally employed as a lubricant for leather and machinery, for greasing wool, and in the manufacture of soft soaps, it being well adapted for the latter purpose.

The marrow fat contained in the bones of the horse is wax-yellow, smeary, and hardens on exposure to the air. It commences to fuse at 149° F., and at 183.2° F. is of the consistency of syrup. It yields a very hard white soda soap.

### *Ruminantia, Cavicornia.*

1. *Butter* (*butyrum; Butter*, G.; *beurre, beurre de vache*, F.) constitutes the oily part of the milk of mammals, but the name is generally restricted to the product obtained from the milk of the cow. The principal constituents of milk are milk sugar, caseine, butter, inorganic salts such as potassium chloride, sodium chloride, calcium phosphate, and water. The average composition of cow butter is:—

| | | |
|---|---|---|
| Butter fat | 3.288 p. c. | 12.524 p. c. solid constituents. |
| Milk sugar and soluble salts | 5.129 " | |
| Caseine and insoluble salts | 4.107 " | |
| Water | 87.476 " | |

Milk is a mixture of extremely finely divided substances (milk globules), insoluble in water, with an aqueous fluid (emulsion). Its specific gravity varies between 1.030 and 1.045.

Seen with the microscope, the milk globules appear mostly round and yellowish, with a dark edge.

When milk is allowed to stand, these globules collect upon the surface and form the cream, beneath which is a bluish, transparent fluid which contains the milk sugar, the salts, and the caseine.

Before the appearance of lactic acid fermentation, which is caused by the fermentation of the milk sugar under the influence of caseine, the deposit of fat on the surface is called "sweet cream," and after the appearance of lactic acid fermentation, whereby the caseine is separated in a jelly, "sour cream." The separation of cream requires from twelve to twenty-four hours. To avoid the loss of time, or to reduce it at least to a minimum, the process of freeing the milk from cream by centrifugal power has been recently introduced. The milk is poured into a large drum of strong sheet metal, and after inserting a few radially arranged divisions which force the milk to participate in the motion, the drum is rapidly revolved (900 to 1000 revolutions per minute), whereby the separation of the milk into cream and skimmed milk is effected in a much shorter time than by the old process. In twenty minutes the cream is deposited next to the shaft of the drum, and the skimmed milk on the periphery. By allowing the drum to gradually come to a stand-still, the layers are deposited over each other and separated.

The manner of preparing butter from the cream varies very much, but the object is always to coalesce, at an appropriate temperature, the fat globules to large lumps by shaking, pounding, or stirring. The process is a purely mechanical one, opinions only differing as regards the effect produced upon the globules. When the butter is "come" it is repeatedly washed with water, which must be often changed, or simply kneaded.

This was formerly done with the hands, but it is more advantageous and cleaner to use a kneading machine, the principal part of which is a grooved roller moving upon a table or a board.

The liquid separated from the butter is called "buttermilk," and consists on an average of 0.24 per cent. of butter, 3.82 per cent. of caseine, 90.80 per cent. of water, and 5.14 per cent. of milk sugar and salts; a large portion of milk sugar, however, having already passed into lactic acid.

Eighteen to 21 parts by weight of milk give 4 parts by weight of cream, from which 1 part by weight of butter is prepared. The proportion of the latter to milk is therefore 4½ to 5 per cent, or, simpler, a cow in good condition gives on an average 1 pound of butter daily.

Fresh butter consists of:—

| | 1. | 2. | 3. | 4. |
|---|---|---|---|---|
| Butter fat | 94.40 p. c. | 93.00 p. c. | 87.50 p. c. | 86.50 p. c. |
| Caseine, milk sugar, extracted constituents. | 0.30 " | 0.30 " | 1.00 " | 0.30 " |
| Water | 5.30 " | 6.70 " | 11.50 " | 13.20 " |
| | 100.00 " | 100,00 " | 100.00 " | 100.00 " |

The effect of the percentage of caseine and water is that the butter soon spoils and becomes rancid. To overcome this, salt is added to the butter in kneading. For 1 pound of butter an allowance of ¼ to ¾ ounce of salt is generally made. In England a mixture of 4 parts of common salt, 1 part of saltpetre, and 1 part of sugar is employed; while for the Danish butter exported in tin cans only saltpetre-water is used. Another means to prevent butter from spoiling is to heat it. The melted butter fat soon rises and is separated from the underlying strata of caseine and water.

Pure butter, freed from all by-constituents, consists of several neutral fats which, as regards their separate quantities, vary considerably in the different varieties of butter, according to the breed of cows, the feeding, season of the year, time after calving, and locality. The larger portion of these fats is a mixture of the general constituents of all animal and vegetable fats.

In butter have been found the glycerides of oleic acid as well as all the members of the fatty acid series to arachidic acid, of which those here printed in capitals form the principal constituents.

| | | |
|---|---|---|
| Acetic acid | $C_2H_4O_2$ | Volatile fatty acids soluble in water; their solubility in water decreases with the increase of atoms of carbon. |
| BUTYRIC ACID | $C_4H_8O_2$ | |
| CAPROIC ACID | $C_6H_{12}O_2$ | |
| CAPRYLIC ACID | $C_8H_{16}O_2$ | |
| CAPRIC ACID | $C_{10}H_{20}O_2$ | |

The last four acids in the form of glycerides constitute the impure butter fat or butyrin, and cause the characteristic odor of butter.

| | | |
|---|---|---|
| Lauric acid | $C_{12}H_{24}O_2$ | traces of the fatty acids volatile and soluble in water. |
| Myristic acid | $C_{14}H_{28}O_2$ | |
| PALMITIC ACID | $C_{16}H_{32}O_2$ | non-volatile fatty acids insoluble in water. |
| STEARIC ACID | $C_{18}H_{36}O_2$ | |
| Arachidic acid | $C_{20}H_{40}O_2$ | |
| OLEIC ACID | $C_{18}H_{34}O_2$ | |

Or, in short, according to the older statements:—

| | | |
|---|---|---|
| Butyromargarine | 66 | per cent. |
| Butyroleine | 28 | " |
| Butyrin | 6 | " |
| | 100 | " |

The average elementary composition of butter fat is:—

| | | |
|---|---|---|
| Carbon | 75.63 | per cent. |
| Hydrogen | 11.87 | " |
| Oxygen | 12.50 | " |
| | | " |
| | 100.00 | " |

and it contains about 1 per cent. less of carbon than the fat of the animal tissue.

There seems further to be present in butter a certain amount of lecithine, $C_{42}H_{84}NPO_9$, a substance widely distributed in the animal organism, occurring in the brain, in the nerves, in the yolk of eggs, etc. It is a wax-like, indistinctly crystalline mass, which readily undergoes decomposition by boiling its alcoholic solution alone, or, more rapidly, in the presence of alkalies or acids, the products being glycerin-phosphoric acid, cholin, and fatty acids, especially oleic and palmitic acids.

There is frequently a yellow coloring matter in butter, very likely due to particular constituents of the feed. It is more pronounced in summer than in fall, while winter butter is whitish-yellow and even yellowish-white. As yellow butter is preferred, it is generally artificially colored with carrot juice, annotto, or safflower, and less frequently with fustic and turmeric.

There are, finally, some other substances in butter, not yet thoroughly investigated, which impart to it its peculiar, agreeable, aromatic taste.

On account of the difference in the composition of butter the melting point varies very much; generally speaking, so-called winter butter is more difficult to melt than summer butter. Below 50° F. butter is hard and crumbling; at 50° to 68° F. sufficiently soft to allow of being readily spread; from 68° to 77° F. quite soft. Fresh butter melts at 88.0° to 89° F. and solidifies with very little development of heat at 66.0° to 68° F.; old butter melts at 89.5° to 91.5° F., and solidifies at 73.5° to 75.0° F., heating thereby to 78° F.

Butter consists originally of an entirely amorphous mass of coalesced fat globules. After melting and cooling it becomes crystalline, the crystallization being more perceptible with the microscope. At ordinary temperatures the specific gravity of butter is 0.920 to 0.940, whereby is to be remarked that the glycerides of the acids rich in carbon (palmitic, stearic, oleic acids) are specifically somewhat lighter than the glycerides of acids poor in carbon, such as butyric, caproic, capric acids, etc.

Natural butter, otherwise of a perfectly good quality, frequently becomes tallowy, *i. e.*, it suddenly shows white streaks, and acquires a perceptible odor and taste of tallow, so that a superficial examination would arouse suspicion of adulteration with tallow. This phenomenon, which thus far has not been sufficiently explained, occurs when butter is exposed, even for a short time only, to the direct light and heat of the sun. On the other hand, it is supposed that butter too much worked or over-salted, the pores of which are not completely filled with brine, but partially with air, readily becomes tallowy.

Variations in temperature are also claimed to be of influence. If butter be kept in a room in which the temperature sinks at times below the freezing point, it is also apt to become tallowy.

In butter made of sour material the phenomenon frequently appears within a few days after the making the butter.

Such butter when examined gives normal numerical values.

Butter fat dissolves readily in ether, with difficulty in cold alcohol, and in 25 parts of boiling alcohol. In testing butter in regard to its purity a conclusion can already be formed from the odor, taste, and color, from the appearance of a freshly-cut surface, the behavior of the butter on pressing with a knife, etc. The odor should be fresh and powerful, but not rancid; the taste pure, agreeably sweetish, aromatic, and not too salty; at an ordinary temperature the cut surface should not be granular and dull, but flexible and somewhat lustrous; pressure with a knife should yield but few drops of water.

The more simple adulterations of butter are with an excessive amount of salt, by means of which a considerable quantity of water can be incorporated, and also with caseine. The percentage of water can be still more largely increased than is the case with salt by an addition of borax and alum, which, however, is readily detected by the taste.

For the determination of water, etc., a determined quantity of butter is placed in a graduated test-tube, which is closed with a good cork reaching exactly to one of the marks, and melted in a lukewarm place. After melting the tube is reversed, *i. e.*, with the cork down, left for a few hours in the lukewarm temperature, and then cooled off. If water be present, the cubic centimetres can be read off below. Should the butter contain other foreign substances, they settle, simultaneously with the water, on the cork end. They are brought together with the water in a tared beaker glass, rinsed with a determined number of cubic centimetres of water, and weighed.

After evaporation the residue is dried at 230° F. and weighed. The loss after deducting the quantity of water used for rinsing, gives the percentage of water; the residue, the total content of caseine and salt, as well as other foreign admixtures, such as gypsum, chalk, heavy spar, potato flour, starch, etc., though the first three exist perhaps more in the imagination of some chemists than in reality.

The residue, extracted with water and dried, gives the caseine; the aqueous solution evaporated, the percentage of salt.

The caseine must be entirely consumed by heat without leaving ash. Should ash be left, the above-mentioned inorganic substances may be present.

As for the further examination of butter, the pure butter fat has to be used; it is necessary to melt it in a lukewarm place. It is advantageous to examine the sediment formed with the microscope, and especially treat it with iodine, which, in the presence of starch, etc., will produce a blue coloring.

The former methods of examining butter fat as to its purity were based upon the solubility of butter in various solvents, such as pure ether, pure alcohol, various mixtures of the two, petroleum-spirit, benzine, etc. But these methods were always unreliable, as mixtures of different kinds of fat might present the same behavior as butter.

The determination of foreign fats is especially directed towards the detection of lard and oleomargarine, etc. Otto Hehner was the first to present a rational method, which, though modified by others, still deserves the preference.

Hehner showed that the fatty acids fixed on glycerin amount to three to four times as much as was generally supposed, and that the quantity of the volatile acids and that of the fatty acids soluble in water are nearly constant in butter fat, or at least vary only within very narrow limits, and are almost independent of the breed of cows giving the milk. Besides the age of the butter to be examined, and whether it be rancid or tallowy,

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exerts no influence whatever upon the results of the examination.

All animal fats, with the exception of butter, consist of tristearin, tripalmitin, and triolein. Now as the atomic numbers of these glycerides are very high, and differ but little from each other, they must by saponification and subsequent decomposition of the soap with hydrochloric or sulphuric acid yield nearly the same percentage of fatty acids; and such is actually the case, the average being 95.50 per cent., tristearin yielding up to 95.73 per cent., tripalmitin to 95.28 per cent., and triolein to 95.70 per cent.

Pure butter fat, however, yields by saponification and subsequent decomposition of the soap only 87 to 88 per cent., and at the utmost 89.73 per cent of insoluble fatty acids, therefore butter with 88 per cent. of such fatty acids may be considered "*pure*," with a higher percentage "*suspicious*," and with over 89.73 per cent. *adulterated.*

The examination of the butter is made by first melting 5 to 10 grammes of butter and allowing it to settle. The fat is then saponified with 2 to 4 grammes of dry caustic soda and a corresponding quantity of water. It will be found advantageous to add a little alcohol, as it promotes saponification, prevents foaming, and produces the agreeable odor of butyric ether. When saponification is complete the soap is evaporated until the alcohol is driven off; it is then dissolved in water and decomposed by hydrochloric or dilute sulphuric acid. After cooling the fatty acids are separated by filtering through a weighed filter paper and thoroughly washed; the paper with the insoluble fatty acids is then thrown into a weighed beaker, dried and weighed, and the percentage calculated.

Reichert's modification of Hehner's method is as follows: 2.5 grammes of the dehrydated fat in a flask of 150 cubic centimeters capacity are heated in a water bath with one gramme of caustic potash and 20 cubic centimeters of alcohol of 80 per cent., and occasionally agitated until frothing no longer takes place and saponification is complete. Fifty cubic centimeters of water are then added, and, when solution is effected, this is followed by 20 cubic centimeters of dilute sulphuric acid (1 volume concentrated sulphuric acid to 10 volumes of water). The contents are now carefully subjected to distillation, a faint current of air being passed through the liquid to prevent bumping, and a ball tube with a wide opening fitted into the retort to prevent the spurting over of any of the sulphuric acid. After 10 to 20 cc. have thus been distilled over, the distillate is returned to the retort, and distillation continued until 50 cc. of *filtered* distillate have been collected. This is tinged with a drop of tincture of litmus and titrated with $\frac{1}{10}$ normal soda solution until the blue color of the litmus is permanent.

As the result of a large number of experiments thus made, Reichert finds that 13 to 14 cubic centimeters of $\frac{1}{10}$ normal soda solution are required for the distillate obtained from pure butter fat. For other fats the following quantities were required; Cocoanut fat, 3.70 cc.; oleomargarine, 0.95 cc.; lard, 0.30 cc.; kidney fat, 0.25 cc.; rape-seed oil, 0.25 cc.

*Modification of Reichert's Method by Dr. B. F. Davenport.**— "Use only 10 c.c. of alcohol in the saponification; the advantage being that with this small quantity the saponification is almost immediate. In fact I begin to draw out the alcoholic vapor from the flask as soon as it comes to a boil, using a water-pump; thus it takes only about fifteen minutes to complete the saponification and the mass evaporated down to a thick mass, free from any alcoholic vapors. My process is to melt the butter at about 80° C.; filter off the clean fat, stir it into a uniform mass as it solidifies. Weigh off two portions (for I work in duplicate). of the solid mass of 5 grams each upon counterpoised double filters of about the size of the scale pan, using double filters that there may be no chance of any thing going through to the scale pan. Roll up the edges of the double filter over the butter upon them, and slip it all into an Erlenmeyer flask. It is easy to get the exact 5 grams upon the open filter, and by putting all into the flask there can be no loss in the transfer. Run off upon the butter in the flask 10 c.c. of 70 per cent. alcoholic solution, containing 2 grams of KOH. Saponify and get dry mass in about fifteen minutes, add to it 100 c.c. of water, dissolve, aided by heat, add 50 c.c. of dilute $H_2SO_4$, containing about one-tenth part of commercial $H_2SO_4$, add several pieces of rough pomace loaded with enough stout platinum wire to lie upon the bottom of the flask, and then distill off 100 c.c. directly into a sugar flask having a small funnel, and filter in its mouth.

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* Department of Agriculture, Div. of Chem. Bull., No. 13. Food and Food Adulterants, Part I., Dairy Products, 1887. (Communicated in Ms. to the Bulletin.)

"I used at first after distilling off about 20 c.c. to pour it back into the flask and then distill off 100 c.c., but I soon learned that that made no difference, as also using a 50 c.c. sugar flask full of diluted $H_2SO_4$, instead of the directed 40 c.c., which was not quite so convenient a quantity to measure off. I leave the end of the bulb tube connecting the flask with the condenser long enough to enter the condenser so far that there is no need of any rubber connection between them, there being no escaping at the upper end of the condenser of any vapor or of scarcely any odor at all. The operation requires no transferring of the material from the beginning to end. Samples of known pure butter have by this method required an average of 28.8 c.c. of alkali to neutralize the 100 c.c. of distillate. I use phenolphthalein as an indicator instead of litmus. When the alcoholic KOH solution has been made for some time I make a blank saponification and distillation with that and discount the cubic centimeters of soda solution required by that for that required by the butters."

There being considerable variation in the iodine number of different varieties of butter, it has been but little utilized for testing butter. Hübl, for instance, found 26.0 to 35.1, an average of 31.0; Moore, 19.5 to 38.0; Woll, 25.7 to 37.9, an average of 32.32 from 56 samples; Williams, 32.25 to 38.91, an average of 35.24 from 30 samples. All animal fats used in the adulteration of butter have higher iodine numbers, but the iodine number of cocoanut oil is 8.9, so that mixtures with the correct iodine number may be readily prepared.

Old, rancid butter is frequently "*worked over*," in order to remove the free acids and the glycerin; the butter is washed with dilute solution of soda or potash, and sometimes with solution of calcium chloride or churned over. Such butter, however, is never fit for table use, though it may be employed for baking or cooking. In examining such butter a higher percentage of insoluble fatty acids, 90.5 to 91 per cent., must be allowed for.

The lower members of the fatty acid series, *i. e.*, those soluble in water and volatile fatty acids, can also be determined in an easy manner. For this purpose a butter soap is prepared, according to Reichert, from 2 grammes of caustic soda and 5 grammes of pure butter fat. After complete evaporation of the alcohol the soap is compounded with dilute sulphuric acid and subjected to distillation. The filtered distillate is titrated with 10 per cent. potash lye, of which never less than 26 to 27 cubic centimeters should be used, as genuine, pure butter demands 27 to 31½ cubic centimeters of the fluid.

Coloring substances intentionally added to the butter can be transferred to dilute spirit of wine or hot water by shaking the butter with one of these liquids. The color of pure butter fat is dissolved in the fat itself and cannot be extracted.

The use of butter is too well known to need further mention here.

2. *Ox tallow or beef suet* (*sebum bovinum; Rindstalg, Ochsentalg*, G.; *suif de boeuf*, F.) from *Bos taurus*, Linn., or the ox. The manner of obtaining and further treatment of tallow have been described (Vol. I, p. 381 *et seq.*), so that only its chemical and physical properties need here be considered.

Ox tallow is hard and solid, pale yellow or white, and, when fresh, without taste or odor. In a short time, however, it acquires a somewhat peculiar odor without being rancid. It is insoluble in cold alcohol, but soluble in boiling alcohol of 0.822 specific gravity, in ether, chloroform, heated benzine, and volatile oils. Fresh, it melts at 108.5° to 109.5° F., and old, at 110° F. It solidifies at 91.5° to 93.2° F., whereby a development of heat to 98.5° and even to 100.5° F. takes place.

The elementary composition of tallow is:

| | | |
|---|---|---|
| Carbon | 76.50 | per cent. |
| Hydrogen | 11.91 | " |
| Oxygen | 11.59 | " |
| | 100.00 | " |

and it contains about 66⅔ per cent. of solid fat-stearin and palmitin, and 33⅓ per cent. of liquid fat-olein. The solid fat is white, granular, and crystalline, melts at 113° to 115° F., and solidifies at 86° F. The liquid fat is colorless and of a very slight odor. This oil has a specific gravity of 0.915 to 0.920 at 59° F., and 123 parts of it dissolve in 100 parts of boiling alcohol.

*Tallow oil*, called in the trade *oleo* (*Talgöl*, G.; *huile de suif*, F.), corresponds to tallow, as lard oil to lard, and is obtained from tallow by pressure. Tallow oil is somewhat thick and pasty, and is well adapted for the manufacture of fine soaps, according to the oolor of the commercial article.

The press residue is the ordinary stearin of commerce.

To completely extract the oleo from the tallow the latter is first melted, and, after nearly cooling with constant stirring, 10 per cent. of its volume of benzine or petroleum-spirit is added. The mixture is allowed to cool in flat sheet-iron boxes, after which the separate cakes are enveloped in cloths and pressed between iron plates in a hydraulic press.

The oleo dissolved in benzine or petroleum-spirit runs off, while stearin and palmitin remain behind. The benzine or petroleum-spirit is then distilled off in a distilling apparatus by the direct introduction of steam, whereby the comparatively pure oleo remains behind.

The solid fats are treated in the same manner to remove traces of benzine, etc.

Ox tallow is frequently compounded with fats of less value, such as fullers' fat, bone fat, etc. This cannot be actually designated a fraud, provided the article be sold under the general name of "tallow," since such an addition only impairs the quality of the product, without being a disadvantage to the buyer who desires a cheap article. But if the consumer orders ox tallow it is no doubt the duty of the dealer to call his attention to the fact that recently much ox tallow adulterated with whale fat is brought into commerce. The melting point of such tallow is considerably lower, and can be recognized by the train-oil odor evolved in slowly melting a sample with common salt.

Adulterations with starch, potato flour, and metallic substances are detected in the same manner as given for lard.

The examination of tallow as to an admixture of other oils may be carried out in the same manner as given for lard. In testing tallow, adulteration with cotton-seed oil has also to be taken into consideration.

However, while with lard Hübl's iodine number is of limited value, it gives safe guiding points in the examination of tallow.

Like with mixtures of lard, the quantity of potassium hydrate required for saponification shows scarcely any variations with mixtures of tallow and cotton-seed oil, as seen from the annexed table:

*Iodine Numbers of Mixtures of Tallow and Cotton-seed Oil.*

| | | | Tallow with | | | | |
|---|---|---|---|---|---|---|---|
| | | | 5 p.c. | 10 p.c. | 20 p.c. | 30 p.c. | 40 p.c. |
| | | | Cotton-seed oil. | | | | |
| Fusing point ° C | — | 43.3 | 43.3 | 42.2 | 40.6 | 39.4 | 39 |
| Per cent. potash for saponification | 19.4 | 19.6 | 19.6 | 19.4 | 19.4 | 19.4 | 19.5 |
| Per cent. iodine for absorption | 109.0 | 40.8 | 44.0 | 47.1 | 52.0 | 59.2 | 66.2 |

| | |
|---|---|
| Saponification number of ox tallow | 193–195 |
| Fusing point of the fatty acids | 114° F. |
| Solidifying point of the fatty acids | 109.4° F. |
| Iodine number of ox tallow | 38–40 |

In commerce two varieties of tallow are known—"candle tallow" and "soap tallow." Russian tallow occupies, without doubt, the foremost place among the foreign varieties.

In the Paris market tallow is sold according to solidifying point of the fatty acids—the *tallow-titer.*

Dalican found the following tallow-titer for tallow and tallow-like fats:

| | | | |
|---|---|---|---|
| Paris tallow | 43.5 | Ox tallow (Odessa) | 44.5 |
| Ordinary ox tallow | 44 | Sheep tallow (Odessa) | 45 |
| Ox tallow (kidney fat) | 45.5 | Tallow from New York | 43.5 |
| Ordinary sheep tallow | 46 | Ox tallow (Buenos-Ayres) | 44.5 |
| Sheep tallow (kidney fat) | 48 | Sheep-tallow (Buenos-Ayres) | 45 |
| Bone fat | 42.5 | Florentine tallow | 44 |
| Russian tallow | 43.5 | Vienna tallow | 44.5 |

According to Schepper and Geitel, tallow and fats which may serve for its adulteration have the following solidifying points:

| | | | |
|---|---|---|---|
| Tallows | 104°–114.8° F. | Bone fat [†] | 105° F. |
| Margarin | 100.4°–111.2° F. | Cotton-seed oil | 104° F. |
| Oleo-stearin | 123° F. | Cotton-seed oil [‡] | 93.2° F. |
| Sheep tallows | 115° F. | Cocoanut oil | 73.4° F. |
| Ox tallows | 112° F. | Stearin grease | 111.2° F. |
| *Suif d'épluchures* [*] | 105°–108° F. | | |

*Method of determining the standard worth* (*titer*) *of tallow.* In buying tallow for soap making it is usual to subject a sample to the test to determine the "titer," or point at which the fatty acids solidify, this being supposed to be a reliable method of ascertaining the stearin contents. Very heavy transactions being based on the results of these tests, it is of great importance that the method adopted by the analyst be one that can be relied on for accurate results. Experience has, however, shown that the methods hitherto in vogue vary so much as to be a matter of serious consideration for both buyer and seller, when they have to be accepted as a basis of large sales or purchases.

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[*] *Suif d'épluchures* is a fat coming into commerce from France which in drying emits an odor of sulphurous acid, shows a green color, and is probably prepared by mixing an animal fat with a poor quality of olive oil.

[†] Contains 4.5 per cent. ash, which is partially present as ash.

[‡] Contains a considerable quantity of a red-brown coloring matter.

On these grounds the council for instruction in analytical chemistry at the Imperial High-school of Technics, Vienna, appointed a committee consisting of Professor Dr. Rudolf Benedikt, of the Imperial Technological Museum, the Imperial Councillor Dr. v. Perger, and Assistant Professor F. Wolfbauer, to investigate a systematic method of analysis proposed by the last named officer, and the committee, after having thoroughly satisfied itself of the superiority and scientific exactness of the method—a conclusion which was further supported by Professor Ferd. Ulzer, who also took part in the investigations, it was ordered that in all future estimates of the solidifying point of samples of tallow, Wolfbauer's method was to be regarded as the only authorized official test.

The method of procedure is thus prescribed by its author: To 120 grains of the sample of tallow, heated in a beaker to only a very little over the fusing point, stir in 45 cubic centimeters of potash lye, made by dissolving 1.25 kilos of pure caustic potash in 1 liter of water (this should have a specific gravity of 1.509). The stirring is continued until a perfectly uniform magma is formed. The vessel covered with a watch-crystal is now placed in a chamber heated to 212° F. After two hours, in the course of which the magma must be several times stirred, it is tested to determine whether the fat is thoroughly saponified or not. This is done by heating a small portion of the soap with 50 per cent. alcohol. If this gives a perfectly clear solution the saponification is complete: if not, the sample must be returned to the hot chamber, and left until the saponification is complete.

The soap when made is decomposed by boiling with dilute sulphuric acid, for which purpose a volume of 165 cubic centimeters of sulphuric acid, of a density of 1.143 = 18° Baumé, is employed. For this purpose a silver vessel is the most convenient, and the boiling is continued until the separated fatty acid floats as a layer of perfectly clear oil on the acid solution of sulphate of potash. (If the silver vessel is large enough to contain more than is prescribed in this process, that is more than 350 cubic centimeters, with allowance for boiling, it is advisable to stir into the soap, before its decomposition, 150 cubic centimeters of boiling water, and to place the mixture in the hot chamber again for ten minutes. The resulting decomposition of the watery soap by the acid proceeds much more rapidly and saves time.) When the decomposition is complete, the silver vessel is covered with a glass saucer containing cold water to guard against any concentration of the acid liquid. Later, the acid solution is completely drained off from the fatty acid and the latter boiled for a quarter of an hour in the covered vessel with a weak solution of sulphuric acid, containing 5 per cent. of concentrated acid. After standing some time, the acid wash-water is carefully drained off and the fatty acids again boiled with about 100 cubic centimeters of water to wash off the last traces of sulphuric acid. When the water, by standing, has separated from the fatty acid, the latter is dried for two hours at a temperature of 212° F. in an open, sufficiently roomy glass vessel, until it is thoroughly dry. If the water on pouring off has an appreciably acid taste, the last process must be repeated, otherwise the fatty acids cannot be regarded as sufficiently dry and pure for exact results.

Next, for the determination of the solidifying point of the fatty acids itself, take a thin-walled test-tube 3½ centimeters in diameter and 15 centimeters long, and fill it with the melted fatty acid to within 1½ centimeters of the rim. The filled test-tube is inserted by means of a cork in a sufficiently large and stable glass bottle, as shown in Fig. 234. The thermometer should be one ranging from 1° to 60° C. and divided into one-fifth of a degree, and should be inserted axially into the test-tube by means of a cork as shown in the illustration.

The cork is not designed to seal the test-tube hermetically. On the contrary, there must be sufficient play to admit of the fatty acids being stirred by the thermometer without difficulty. In order that the thermometer may be as short as possible, it is provided with a bulbous crop between +2° and 28°, by which means the scale appears to be shortened about 28°, as shown in

FIG. 234.

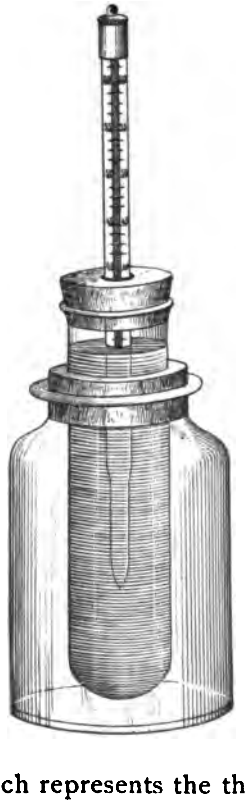

FIG. 235.

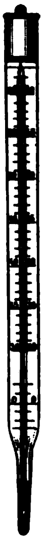

Fig. 235, which represents the thermometer one-third its actual size. By this means it is possible to prevent too great a length of the column of mercury standing above the fatty acid, and thus guard against an important possible source of error (the lowering of the solidifying point). By this arrangement the thermometer is submerged in the fatty acid mass to about 35°, which is sufficient to guard against error.

The stearine mass is stirred with the thermometer until the previously clear mass has become quite opaque, and in fact until the mass begins to solidify. At this point the thermometer stops sinking, and remains constant even if stirring at a moderate speed ten times in a circle. The stirring must now cease, and the thermoneter being left to itself will begin to rise in consequence of the liberation of the fusing heat of the solidifying fatty acid. The highest point, which as a rule persists for some minutes, is to be taken as the solidifying point. It is, of course, essential that the thermometer be exact, and it is well to examine the zero point occasionally.

The employment of narrower test-tubes than that described leads to less reliable results, and so also the neglect of observing the fact that by passing from a fluid into a solid aggregate state a so-called over-cooling takes place with fatty acids as well as with neutral fat.

Every such test to determine the solidifying point must invariably be repeated, and has to be regarded as faulty if the difference in the results exceeds 0.1° C. As a rule the difference will not exceed 0.05°. It is scarcely necessary to remark that the fatty acid used in determining the titer has to be tested with alcohol and ammonia as to its degree of saponification.

To test the exactness of the method here given, and also to determine the extent to which the variations in the process influence the final results, *i. e.*, the solidifying point, numerous experiments have been made, and this with a carefulness which admits of their being accepted as quite reliable.

The following are the data arrived at:

(1) To determine the "standard" it is immaterial whether saponification is effected by means of alcohol or acoholic lye, provided equal care is taken in washing out both solvents. It may be remarked that for the thorough removal of the alcohol the soap solution, thinned with water, requires to be boiled one and a half hours.

(2) It is a matter of no consequence whether the tallow with the alkaline lye be kept heated and stirred 1½ hours or 15

hours. The lengthening of this stage of the process is hence immaterial.

(3) That the amount of water in the fatty acids exercises a most important influence upon the results, the most important variations in the solidifying point of any given sample of fatty acids being exhibited when a portion is tested without drying, a second portion partially dried, and a third portion fully dried. Filtering removes part of the water or alcohol, but gives no reliable results.

(4) The employment of a narrower test-tube than that prescribed results in a lowering of the solidifying point. A test tube 2½ cm. diameter gave a mean depression of the solidifying point equal to 0.2° Celsius. On trial with a larger test tube, viz., 7 cm. diameter, and containing four times the amount of the prescribed test-tube, the results were not affected. It may hence be taken as proved that a 3½ cm. test tube suffices for reliable results.

(5) As regards the thermometer, it is important that the column of quicksilver do not rise higher than the medium in which it is submerged, for if it rise into a cooler medium it will be sensibly lowered. A thermometer with a reduced scale, such as is here recommended, is consequently necessary to an exact determination of the point of solidification. In a most carefully conducted experiment with the longer thermometer first tested by the thermometer in use, and agreeing perfectly with it when both were submerged, the longer thermometer with its mercury raised above the fatty acid marked a reduction of 0.12° in the solidifying point.

(3) *Beef marrow* (*Rindermark*, G.; *moelle de boeuf*, F.), is contained in the bones of the ox, and possesses nearly the same properties as tallow. In fact, the marrow of all mammals has nearly the same characteristics as the fat from the other parts of the same animal, the difference in the taste between melted beef marrow and ordinary fat being due to foreign substances in the vessels surrounding the tissue of the adipose cells.

Beef marrow melts at 113° F., and solidifies at 95° F., developing thereby heat up to 104° F. After cooling it shows a granular structure.

It consists of about 70 per cent. of palmitin and stearin, and 30 per cent. of olein, dissolves incompletely in boiling alcohol, and with more difficulty in ether than ox tallow. It also becomes rancid sooner than the latter. It is chiefly used for pomades,

(4) *Sheep's tallow or mutton suet* (*sebum ovile*; *Hammeltalg*, G.; *suif de mouton, graisse de mouton*, F.), is obtained from *Ovis Aries*, Linn., or the sheep. It resembles ox tallow, but is whiter, somewhat harder, and brittle. Fresh, it is odorless, but on exposure to air acquires in a short time the characteristic sheep odor and taste, and soon becomes rancid, it behaving in the latter respect similar to *goat's tallow* from *capra domestica*, Linn., the goat, which possesses a characteristic goat odor. According to Chevreul, these odors are due to hircinic acid which, however, is very likely only a mixture of butyric acid, with other volatile fatty acids, etc.

Fresh sheep's tallow melts at 115° F. and solidifies at 97° F., becoming heated thereby to from 104° to 106° F. Old sheep's tallow fuses at 120° to 122° F. and solidifies at 102° to 104° F., becoming heated thereby to from 111° to 113° F. After cooling the fat shows a smooth surface, and in the interior traces of a crystalline structure. It is soluble in more than 60 parts of cold ether and in 45 parts of boiling alcohol of 0.821 specific gravity.

The elementary composition of sheep's tallow is:—

| | | |
|---|---|---|
| Carbon | 76.61 | per cent. |
| Hydrogen | 12.03 | " |
| Oxygen | 11.36 | " |
| | 100.00 | " |

It consists of about 70 per cent. of stearin and palmitin (margarin) and 30 per cent. of olein.

The olein obtained by pressure is colorless, has a slight mutton odor, and a specific gravity of 0.913 at 59° F. Eighty parts of it dissolve in 100 parts of boiling alcohol.

On account of the crystalline appearance of the stearin obtained from sheep's tallow, it is preferred to ox tallow for the manufacture of candles. Frequently both varieties of tallow melted together are found in commerce.

### *Aves. Palmipedes.*

*Goose grease* (*Gänsefett*, G.; *graisse d'oie*, F.) is obtained from *Anas anser domesticus*, Linn., or the goose. It is of a very pale yellow color, of an agreeable taste and odor, and of a soft and somewhat granular consistency. It fuses at 77° to 79° F. and solidifies at 64.5° F., whereby it becomes heated to 71.5° F. By pressure at 28.5° F. it can be separated into 32 per cent. of grease, which melts at 111° F., and 68 per cent. of a slightly colored oil.

By saponification goose fat, besides glycerin, gives, stearic, palmitic, oleic, caproic, and butyric acids. It contains on an average 2.94 per cent. of soluble and 92.93 per cent. insoluble fatty acids.

## 5. *Non-Glycerides or Waxes.*

### A. WAXES OF THE VEGETABLE KINGDOM.

### *Cassuvieæ* (*Terebinthinæ*).

*Japanese wax* (*cera japonica; Japanisches Wachs*, G.; *cire du Japon*, F.) is chiefly derived from *Rhus succedanea*, Linn., *Rhus vercinifera*, De C., and from *Rhus sylvestris*, the first two being indigenous to China and Japan, and the latter to Japan only. To obtain the wax the usual method is as follows: The fruits are ground, either with or without the stones, by means of millstones, and the floury mass boiled with water in large kettles and the wax skimmed off. To remove foreign admixtures it is several times remelted until clear. By another process the fruits are first beaten with bamboo flails, then dried for fourteen days, slightly roasted, and ground by means of millstones. The floury mass is exposed in small bags to the action of water vapor to melt the fat in the cells, which is then expressed by means of different presses. The extraction process has recently also come into use. The expressed wax being somewhat greener than that extracted with ether or carbon disulphide, it is boiled with dilute lye, whereby it becomes granular and more susceptible to the bleaching process, then washed with water and exposed to the sun. According to the degree of bleaching the wax is known as prime and second.

To obtain the last remnants of wax from the press residue about 10 per cent. of perilla oil* or "*ye goma*" is frequently added during the second pressing in order to retard solidification. Such an addition exerts, of course, an influence upon the consistency of the wax, this being the reason that hard and softer varieties are found in commerce.

Japanese wax is now brought into commerce in square cakes weighing about 140 pounds each. The mass has a waxy appearance, is whiter and more brittle than beeswax, and shows an even or largely conchoidal fracture. The surface of a fresh fracture is lustreless; that of a cut one has, however, a wax-like lustre. The wax can be kneaded between the fingers and chewed to a powder. Its odor and taste are somewhat resinous, tallowy and rancid. By storing it becomes yellow to brownish, and when it contains much water becomes covered with a white, dust-like film.

Viewed under the microscope the interior of the wax consists of small and large refractive grains, between which are small needle-like crystals of palmitic acid. The yellow exterior mass presents a like condition, only that it contains more crystals than the interior. The white coat is rich in small bar-like and broad prismatic crystals.

Japanese wax fuses at 128° to 130° F. and solidifies at 105° to 106° F., whereby it becomes heated to from 114° to 115° F. In heating the wax shows the peculiarity of becoming transparent at 18° to 23.5° F. below its fusing point, and to this peculiarity, and that of some fats acquiring a higher fusing point by long storing, are no doubt due the various statements as regards the fusing point, which has been given by

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* From *Perilla ocimoides*, Linn.

| | |
|---|---|
| Hanbury at | 125.6° to 131° F. |
| Oppermann at | 120° to 122° F. |
| Stahmer at | 107.6° F. |
| Trommsdorff at | 117.5° F. |
| Müller at | 107.6° F., etc. |

If Japanese wax is remelted a short time after solidifying, it fuses at 107.5° F., and requires the ordinary fusing point of 128° F. only after long storing. This may be another reason for the varying statements about the fusing point.

The specific gravity of the crude wax is 1 to 1.006, and that of bleached wax 0.970 to 0.980 at 59° F.

Japanese wax is insoluble in cold alcohol, but readily so in boiling 97 per cent. alcohol. After cooling the solution solidifies to a white, granularly crystalline mass. It is insoluble in cold ether, but readily soluble in warm ether; on cooling it is, however, nearly all again deposited in flakes. It is easily soluble in benzine and petroleum-ether.

The designation "wax," which has been applied to this body, is not appropriate, since it is not an actual fat, it containing no olein, and, on the other hand, it is not an actual wax because it is a glyceride. It consists chiefly of the glyceride of palmitic acid, the corresponding compounds of stearic acid, and especially of arachic acid, amounting only to 6.2 per cent. Besides the glyceride the commercial article also contains crystals of free palmitic acid.

Japanese wax is composed of:—

| | East India wax. | West Indies wax. | Wax purified with ether. |
|---|---|---|---|
| Carbon | 70.00 p. c. | 71.88 p. c. | 73.12 p. c. |
| Hydrogen | 12.07 " | 12.00 " | 11.85 " |
| Oxygen | 17.93 " | 16.12 " | 15.03 " |
| | 100.00 " | 100.00 " | 100.00 " |

It is remarkable for its small percentage of ash (from 0.02 to 0.08 per cent.) and the ease with which it saponifies.

It is frequently adulterated with as much as 30 per cent. of water and tallow. Water is detected in the same manner as in butter, lard, etc., by carefully melting a sample and allowing it to settle in a lukewarm place, and tallow by the lower melting point and the solubility in alcohol, ether, etc., but in the most simple and quickest way by the odor of acrolein.

In Japan the wax is used as a substitute for tallow and beeswax in the manufacture of candles, and also for producing a lustre upon turned wooden articles. In Europe it is employed in the manufacture of wax matches, as an ingredient in polishing materials for shoemakers' and cabinetmakers' use, and as an addition to bleached-beeswax in the manufacture of candles, as it facilitates the removal of the latter from the moulds. For pharmaceutical purposes it is not well adapted on account of its rancidity, which promotes the rapid deterioration of fats mixed with it. Perfumers use it for preparing a castor-oil pomade, a mixture of castor-oil and Japanese wax, having the property of becoming transparent by frequent remelting.

Among the varieties of vegetable wax, Japanese wax forms the most important article of commerce. The principal European market is London.

### *Myristiceæ* (*Trisepalæ*).

*Ocuba wax* (*Ocubawachs*, G.; *cire d'Ocuba*, F.) is obtained from the fruit of *Myristica Ocuba*, Humb. and Bonpl., a bush indigenous to Brazil and Guiana. The fruit is the size of a small hazelnut and the seed is covered with a purple coating. The fruit is comminuted and boiled with water, whereby the wax separates on the surface and is skimmed off after cooling. Yield 18 to 20 per cent.

Like Japanese wax Ocuba wax does not belong to the actual waxes, it being a mixture of wax, fat, and resin. It is yellowish white, softer than beeswax, has a specific gravity of 0.920 at 59° F., melts at 102° to 104° F., and is sparingly soluble in cold alcohol, but completely so in boiling alcohol and ether. In the manufacture of candles it can only be used mixed with other materials, its fusing point being too low.

### *Artocarpeæ* (*Urticinæ*).

1. *Ficus wax*, *fig wax* or *Getah wax* (*Feigenwachs*, G.; *cire de figuier*, F.) is obtained from *Ficus ceriflua* or *Ficus cerifera*, Blume, or the wax-fig tree, indigenous to West and Central Java, Sumatra and Ceylon. The wax is obtained by evaporating the milky juice of the tree over an open fire and subsequent treatment with water. It has a reddish-brown color, but becomes pure white by bleaching. It is very brittle and friable, softens at 113° F., and fuses at from 133° to 134.5° F. It is partly soluble in alcohol and completely so in ether, petroleum-ether, and boiling alcohol. It is used like beeswax in the manufacture of candles.

2. *Cow-tree wax* (*Kuhbaumwachs*, G.) is obtained from *Galactodendron utile*, Kunth, or the cow tree, which grows in the mountains of South America, especially in the neighborhood of Burbula. By making incisions in the trunk and limbs a milky juice of a pleasant taste, but disagreeable odor, is obtained, which in color and taste resembles cow-milk, but differs from it in being of greater consistency and containing some admixtures of vegetable mucus. It does not curdle with acids, and is free from the acridity of other vegetable milky juices. It contains 30 to 35 per cent. of wax, which is separated by boiling.

Cow-tree wax is somewhat transparent, can be kneaded, fuses at 122° to 125.5° F., and can be partly saponified. In external appearance it resembles beeswax more than any other vegetable wax, and yields very good candles, which burn with a bright flame.

### *Myricaceæ* (*Myriceæ*).

*Myrica wax* or *myrtle wax* or *bay-berry tallow* (*cera myricæ; Myricawachs*, G.; *cire de myrica*, F.) is obtained from *Myrica* *cerifera*, Linn., a fragrant shrub which grows near Lake Erie, but chiefly along the Atlantic coast of North America. The fruit secretes wax, which is deposited in thin layers, and covers it with a snow-white crust. This crust is interspersed with small brown or black specks, and can therefore not be removed in its entirety, but only in the form of a powder.

The method of obtaining the wax is very simple. The berries are boiled in water and the fatty mass collecting on the surface is skimmed off and poured into shallow vessels. The yield is about 20 to 25 per cent., or 2 pounds of wax from 8 to 10 pounds of berries. Each bush yields about 20 to 30 pounds of berries.

Myrica wax, which is brought into commerce from the United States, has a deep green color, which, by exposure to light and air for several years, changes to a gray yellow, but only a few millimeters below the surface. It breaks with a shallow, conchoidal fracture.

The color of the wax is due to chlorophyl. Freshly-fractured surfaces become covered, on exposure to the air, with a white film. The taste and odor of the wax are slightly balsamic, but are, nevertheless, so tempting to the Hottentots that they eat it like cheese.

The specific gravity of the wax is 1.000 to 1.005 at 59° F. and it fuses at 113° to 115° F. It is very sparingly soluble in cold alcohol and ether. Hot alcohol dissolves it, but deposits four-fifths of it on cooling. Four parts of boiling ether dissolve one part of wax, but on cooling most of the wax is again precipitated, while the ether acquires a beautiful green color. Cold turpentine only softens the wax, while hot turpentine dissolves about six per cent. It is more readily soluble in fat oils, and easily saponified with alkalies.

Myrica wax contains much palmitic acid, with little myristic and stearic acids largely in a free state, a smaller portion being fixed on glycerin. It contains no oleic acid. The content of ash amounts to about 0.17 to 0.20 per cent.

Myrica wax is used like beeswax, but being less ductile and plastic is generally mixed with it. In America it is much liked for the manufacture of candles, as they diffuse an agreeable odor when extinguished.

### *Balanophoreæ* (*Aristolochieæ*).

*Balanophore wax* (*Balanophorenwachs*, G.; *cire de Balanophore*, F.) is obtained from *Langsdorffia hypogæa*, Mart., and the *Balanophora species*, the first indigenous to Brazil and the latter to Java and the neighboring islands. The wax fills the parenchymatous cells and the plants are so rich in it that when ignited they burn with a bright flame. By converting them into a sort of paste and coating wooden sticks with it, the latter can be immediately ignited and used as torches. The wax is obtained by boiling the plants in water. Thus obtained it is grayish-yellow, while that extracted with ether has the color and consistency of natural beeswax. It is sparingly soluble in alcohol, but readily in ether, melts at 212° F., and has a specific gravity of 0.995 at 59° F. It has the peculiarity of dissolving in cold sulphuric acid and being precipitated from the solution by water.

This wax, actually a resinous wax or waxy resin, is partly a glyceride. It is so far of but little commercial value.

### *Palmæ* (*Spadicifloræ*).

*Carnauba wax* (*Carnaubawachs*, G.; *cire de Carnahuba*, F.) is obtained from *Corypha cerifera*, Linn., or the carnauba palm, which grows on the shores of the rivers of Brazil. The leaves, in the axillæ of which the wax is secreted in the form of scales, are collected and dried. By shaking or pounding the leaves the wax peels off, and is then melted over an open fire or boiled with water, whereby the foreign substances settle on the bottom and the wax collects on the top.

Crude carnauba wax is dirty gray-yellowish to greenish, hard, brittle, and can be readily rubbed to a powder. It is tasteless, and when fresh smells like new-mown hay, but later on becomes inodorous. It is purified by remelting, and has then a greenish-yellow color which cannot be removed.

Fresh purified carnauba wax melts at 185° to 187° F., old wax at 194° to 196° F., and solidifies at 187° to 188.5° F., the cooled mass being somewhat crystalline. In melting the wax diffuses a slight but not disagreeable odor, and by destructive distillation yields a paraffine-like product. Its specific gravity is 0.995 to 0.999 at 59° F.

It is partly soluble in cold alcohol, but completely so in boiling alcohol and in ether. The concentrated solutions solidify on cooling, separating a white crystallizable mass which fuses at 221° F.

Carnauba wax is used to impart to fatty bodies, ceresin, paraffin, wax, etc., a certain degree of hardness and a peculiar lustre, as well as to raise their fusing points. By an addition of only 5 per cent. of carnauba wax to one of the above-mentioned substances, the fusing point is considerably increased; further additions of a like quantity raise the fusing point, only in a constantly decreasing degree. The same is the case in increasing the hardness and lustre.

One gramme of carnauba wax requires for saponification 94.5 to 95 milligrammes of caustic potash; hence, since stearic acid requires 197, beeswax 100.4, Japanese wax 122, paraffin and ceresin each 0 milligrammes of caustic potash for 1 gramme of substance, this property may serve for the quantitative determination of carnauba wax in mixtures with the above-named substances. Light may also be thrown upon the nature of such mixtures by determining the fusing point, as shown by the following table:

### *Fusing point of carnauba wax with stearic acid, paraffin and ceresin.*

Mixtures of carnauba wax = 185° F. fusing point.

| In the proportion of | With stearic acid, fusing point=137.3° F. | With paraffin, fusing point=140.2° F. | With ceresin, fusing point = 163° F. |
|---|---|---|---|
| | Fusing point of the mixture. | Fusing point of the mixture. | Fusing point of the mixture. |
| 5:95 | 157.2° F. | 165° F. | 174.2° F. |
| 10:90 | 164.75° " | 174.3° " | 177° " |
| 15:85 | 166.2° " | 178° " | 179° " |
| 20:80 | 167.3° " | 178.7° " | 180.6° " |
| 25:75 | 168.4° " | 179° " | 181.3° " |

By boiling with potash lye carnauba wax acquires a reddish color; it saponifies only partially and with difficulty with alcoholic potash lye.

H. Stürke has determined in carnauba wax the following combinations:

1. Hydrocarbon; fusing point, 138.2° F.
2. An alcohol $C_{23}H_{56}CH_2OH$; fusing point, 168.8° F.
3. Myricylic alcohol, fusing point, 186° F.
4. Diatomic alcohol $C_{23}H_{46}(CH_2OH)_2$; fusing point 218.3° to 218.4° F.
5. An acid (isomeric with lignoceric acid) $C_{23}H_{47}COOH$; fusing point, 162.5° F.
6. An acid (isomeric or identical with cerotic acid) $C_{26}H_{53}COOH$; fusing point, 174.2° F.
7. An acid $\substack{CH_2OH \\ COOH}$ or its lacton $C_{19}H_{33}{<}\substack{CH_2 \\ CO}{>}O$, fusing point, 218.3° F., from which has been prepared a dicarbonic acid $C_{19}H_{38}(COOH)_2$ with a fusing point of 194° F.

Carnauba wax is composed of:

| | | |
|---|---|---|
| Carbon | 80.33 | per cent. |
| Hydrogen | 13.07 | " |
| Oxygen | 6.60 | " |
| | 100.00 | " |

It is extensively used in the manufacture of candles and varnish.

Formerly large quantities of carnauba wax were brought to England, where they were purified by re-melting. At present it is shipped in a purified state from Brazil to Europe, and forms a not unimportant article of commerce, the value of the wax produced annually in Brazil amounting to over $250,000.

Carnauba wax is suitable for the manufacture of candles which are to be colored. For white candles it must, however, be decolorized, which is effected with bone black, hydrogen peroxide, or potassium permanganate.

Decolorization by means of bone-black is effected by heating the wax in an iron kettle to 230° F. and allowing it to run through a layer of coke into the filter. The latter should be about 8 feet high and 10 inches wide, and be provided with a jacket for heating by steam. Three passages through the filter generally suffice for decolorization. It is also customary to add to the melted wax 10 per cent. of finely elutriated bone-black and, after stirring continuously for 2 hours, to pass the mass through a filter.

For bleaching with hydrogen peroxide the wax is melted in a kettle and allowed to fall through an aperture upon a grooved plate revolving with great velocity, whereby the wax is converted into fine dust and falls into cold water. This finely divided wax is dried before use and brought into 5 or 6 per cent. hydrogen peroxide, which has been saturated with ammonia or sodium carbonate and heated to 122° F. By adding 5 per cent. of borax to the fluid, decolorization proceeds more rapidly and more uniformly.

The decolorized wax is used in the manufacture of candles which are hard and run very little. Generally 20 per cent. stearin or ozocerite (frequently both) is added to the wax. Thus the candles of an English factory consist of carnauba wax 60 parts, stearin 25, ozocerite 15.

*Brilliant paraffin* consists of paraffin 75 parts, carnauba wax, 25.

*Encaustic varnishes* with carnauba wax are more brilliant than those prepared with ordinary wax. Such a mixture consists of water 10 quarts, sodium carbonate 2½ lbs., carnauba wax 5½ lbs.

*Carnauba varnishes* are obtained, 1st, by saponifying carnauba wax with potash solution to which a small quantity of caustic potash has been added; 2d, by decomposing this wax-soap with a metallic salt, such as alum, blue vitriol, green vitriol or chloride of manganese; 3d, by dissolving the precipitate obtained in benzine, oil of turpentine or petroleum.

Finally it may be remarked that Edison's phonograph-cylinders are largely made of carnauba wax.

*Palm wax* (*ceroxylin*) (*Palmwachs*, G.; *cire de palmier*, F.) is obtained from *Ceroxylon andicola*, Humb. et Kunth., or the common wax palm, and from *Klopstockia cerifera*, Karsten, or the Klopstock palm, the first indigenous to the West Indies and the Andes of South America, and the latter chiefly to New Granada.

The trunk of both varieties secretes a resinous wax, which, when scraped off, forms a gray-white powder, each tree yielding an average of twenty-five pounds. The wax thus obtained is converted into a compact mass by melting over an open fire, and purified by remelting.

By another process the bark of the trees is boiled in water, whereby the wax, however, does not melt, but only softens, while the admixed impurities settle on the bottom.

The wax thus obtained presents a yellow or yellowish-white mass, and is brought into commerce either in irregular lumps or balls. In hardness and brittleness it corresponds to carnauba wax. It breaks with a conchoidal fracture.

Both the crude and purified wax have a specific gravity of 0.992 to 0.995 at 15° F., and though they become soft by the heat of the hand, melt only at 215.5° to 221° F.

Palm-tree wax is a mixture of wax and resin, from which, by dissolving in much boiling alcohol, the wax is separated as a white jelly, which, however, is somewhat crystalline. By repeated washing and recrystallizing from a large quantity of boiling alcohol it is entirely freed from resin. This pure wax is white-yellow, resembles beeswax, and melts at 161.5° F. to a but slightly colored liquid. It consists of ceryl cerotate and myricyl palmitate, with resin. It is used in the manufacture of candles, mixed with a little tallow to make it less brittle. The so-called palm candles have almost a lemon color, as palm-tree wax cannot be bleached. Palm wax is but seldom met with in European commerce.

## B. Waxes of the Animal Kingdom.

### *Anthophilæ* (*Hymenoptera*).

*Beeswax, or yellow wax* (*cera citrina; Bienenwachs*, G.; *cire d'abeilles*, F.), from *Apis mellifica*, Linn., or the bee, which yields most of the wax found in commerce, but also from allied species, such as *Apis fasciata*, indigenous to South America, and *Apis unicolor*, found in Madagascar. The wax is secreted in glands placed upon the ventral scales of the bee. With this wax the bees construct the comb, the cells of which are hexagonal with angular bottoms. The substance called "*propolis*" is collected by the bees from the buds of trees. It is of a resinous nature, and is used for lining the cells of a new comb, stopping crevices, etc.

The comb from which the honey has been allowed to drip is first subjected to pressure. It is then melted in boiling water to free it from adhering honey and other impurities, and then poured into flat moulds previously moistened and left to cool.

In a pure state beeswax is perfectly white, but becomes colored by the contact with honey and pollen, the color of crude wax being more or less yellow, and sometimes even reddish or greenish, according to the material used by the bees. It has a remarkable, peculiar, and agreeable honey-like odor, is almost tasteless, does not stick to the teeth in chewing, and is not unctuous to the touch. Its specific gravity is 0.960 to 0.963 at 59° F., wax from tropical countries being somewhat heavier, but never over 0.966. In the cold it is brittle and hard, and, if not adulterated with tallow, will bear chalk marks. At ordinary temperatures it is firm and breaks with a dry, granular fracture. It softens in the hand, fuses at 143.5° to 144.5° F. to a clear, oil-like liquid, and solidifies again immediately below the fusing point without development of heat. It yields nothing to cold alcohol, but nine-tenths of it are soluble in hot alcohol. It dissolves completely in 10 parts of hot ether, in hot volatile and fat oils, and in benzine, carbon disulphide, and chloroform.

It consists of—

| | | |
|---|---|---|
| Carbon | 79.30 p. c. | 79.83 p. c. |
| Hydrogen | 13.20 " | 13.44 " |
| Oxygen | 7.50 " | 6.73 " |
| | 100.00 " | 100.00 " |

By saponification it yields 46 to 47 per cent. of saponifiable and 53 to 54 per cent. of non-saponifiable substances. It consists of 10 per cent. of ceryl cerotate $\left.\begin{matrix}C_{27}H_{53}O\\C_{27}H_{55}\end{matrix}\right\}O$ insoluble in boiling alcohol, and 90 per cent. of myricyl palmitate $\left.\begin{matrix}C_{16}H_{31}O\\C_{30}H_{61}\end{matrix}\right\}O$ soluble in boiling alcohol, though these percentages are not constant.

As by-constituents it contains coloring and odoriferous substances, and a resinous yellow coloring matter which, by dissolving yellow wax in hot alcohol and subsequent cooling, remains in a state of solution.

Wax boils at 459° F., and by destructive distillation yields first a colorless aqueous distillate, "*wax spirit*," then a thickly fluid oil, "*wax butter*," which generally solidifies in the neck of the retort, and, finally, a yellowish, thinly fluid, empyreumatic oil, "*wax oil*." These products were formerly employed in medicine for external and internal use.

Yellow wax is chiefly used for technical and pharmaceutical purposes.

Large quantities of wax are produced in the United States, that from New York being the best quality, while that from the Southern States is inferior, as it cannot be thoroughly bleached. South American wax, "*Andaquia wax*," and that from the Antilles, is of an entirely different color, and is chiefly brought into commerce from Hayti. As in African wax, from *Apis unicolor*, there is more loss in melting and the bleaching process is much slower than with American wax, though the product obtained is of good quality and thoroughly bleached.

All European countries produce wax, most of it coming from Germany, France, Italy and Russia.

India furnishes a gray-brown wax of a slight odor, which is mostly exported to China, the latter country, though producing much wax, consumes it all itself.

Wax from Senegal is of very poor quality, frequently almost black and impossible to bleach. Abyssinia produces a fair quality of wax which is is in much demand.

The principal use of wax is in the manufacture of candles; but yellow wax is not well adapted for this purpose, as candles made from it burn badly and give a yellow, sooty flame. It must therefore be subjected to purification and bleaching, and forms, then,

*White or bleached wax.*—For purification, the already mentioned process of melting the wax in hot water is repeated until it is freed from all honey and shows no longer a gray color.

The destruction of the coloring matter by chemical means is not advisable, as chlorine and other bleaching substances exert a decomposing effect upon the wax, making it brittle and crummy, which has to be remedied by an addition of tallow. The use of chlorine is further objectionable on account of the formation of products, which by combustion develop hydrochloric acid.

The best method of bleaching is to melt the yellow wax in a large vat by means of steam. It is then run off, while in a melted state, into a trough called a cradle, which is perforated at the bottom with holes and placed over a large water tank, at one end of which is a revolving cylinder almost immersed in water. By this means the wax is solidified, converted into a kind of ribbon, and conveyed on the surface of the water to the other end of the tank. These ribbons of wax are lifted out and carried in baskets to the bleaching ground, where they are exposed to the air for one or two weeks, according to the weather, being turned every day and watered from time to time.

By another process the wax, purified by steam, is obtained in threads or grains by allowing it to drop in a thin stream or drops into ice-cold water from a revolving tinned copper boiler provided with one or more cocks. By this granulating and thread-drawing process a greater surface is exposed and the bleaching process shortened at least one-third.

After exposure upon the bleaching grounds the wax is remelted in hot water, strained and poured into moulds to cool. The loss in weight caused by bleaching is from two to ten per cent.

To assist the natural or sun-bleaching process, which is based upon the formation of ozone and the action of the latter upon the coloring matter, by a further formation of ozone, the yellow wax is melted together at a moderate heat with rectified oil of turpentine in the proportion of 8 parts of wax to 1 to 1½ parts of oil of turpentine. The mixture is melted, ribboned, and bleached in the same manner as wax. The bleaching process is much accelerated, being finished in five to six days, after which the odor of oil of turpentine has entirely disappeared.

Wax bleachers frequently add various substances, such as tartar, alum, white arsenic, etc., to promote bleaching. Such additions, besides being of no value whatever, must be removed by remelting the wax with water. Some bleachers add to the wax a certain quantity of suet, from 3 to 5 per cent. There are several reasons for this, the principal being that pure wax when bleached is too brittle; moreover, in the presence of a small quantity of suet, the bleaching goes on more rapidly, and without this suet it is difficult to obtain a product entirely white.

The methods of bleaching wax by means of chlorine, bichromate and permanganate of potash, are the same as those employed for oil, and will be mentioned later on.

The composition of waxes bleached in various ways is illustrated in the following table:

| | Melting point °F. | Free acids in mg. KOH per g. | Total acids in mg. KOH per g. | Iodine absorbed. | Volume of H at °0 and 760 mm. furnished by g.wax. | Hydrocarbons. |
|---|---|---|---|---|---|---|
| | | | | Per cent. | C.c. | Per cent. |
| Pure yellow wax....... | 146.3 | 20.17 | 93.49 | 10.87 | 53.0 | 13.54 |
| Wax bleached in air with 5 per cent. terebenthine............... | 146.3 | 20.20 | 100.40 | 6.78 | 54.9 | 12.39 |
| Wax bleached with oxygenated water....... | 146.3 | 19.87 | 98.42 | 6.26 | 56.1 | 12.53 |
| Pure yellow wax....... | 145.4 | 20.40 | 95.06 | 11.23 | 54.5 | 14.30 |
| Wax bleached with animal black .......... | 145.4 | 19.71 | 93.20 | 11.36 | 53.6 | 13.30 |
| Wax by permanganate | 146.7<br>146.3 | 22.63<br>21.96 | 103.29<br>99.24 | 2.61<br>5.80 | 53.6<br>55.5 | 13.30<br>13.34 |
| Wax by bichromate.. | 145.7<br>147.2 | 21.86<br>23.43 | 98.90<br>107.72 | 7.94<br>1.08 | 51.0<br>53.6 | 13.24<br>11.77 |

The composition of waxes decolorized by chemical processes, as is seen from the table, is quite variable. Chlorine bleaches waxes, but the chlorine is absorbed as iodine and bromine are, and thus the constitution of the wax is profoundly modified. Granulated wax is readily bleached by placing it in a bath of hydrogen peroxide.

White wax is tasteless and inodorous, translucent on the edges, brittle, not unctuous to the touch, softens at 86° F. and in the hand by kneading, melts at 145.5° to 147° F., behaves on solidifying like yellow wax, and has a specific gravity of 0.965 to 0.969. Its solubility in the various solvents and its chemical and elementary composition are nearly the same as those of yellow wax.

In buying wax great care should be exercised, as both the

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yellow and white varieties are frequently adulterated. The specific gravity and melting point are of special importance for the determination of quality.

The most common adulterations are as follows: *Admixture of water* to increase the weight, which is already recognized by the dull and rough fracture and by slowly heating to the fusing point and cooling, whereby the water separates, the quantity being determined by weighing the cooled wax. *Additions of pulverulent substances*, such as *yellow ochre*, *brick meal*, *pea flour*, *heavy spar*, *clay*, *litharge*, etc., are also detected by melting, whereby they separate. *Stearin*, *resin*, and *vegetable* waxes increase the specific gravity. Pure white wax floats in the centre of a fluid consisting of 2 parts of alcohol of 0.830 to 0.831 specific gravity and 7 parts of distilled water, even if the fluid is brought to a specific gravity of 0.965 to 0.970 by the addition of water.

Pure yellow wax shows the same phenomenon in a fluid of 0.955 to 0.965 specific gravity prepared as above from 1 part of alcohol and 3 parts of water. Adulterations of wax with the above-mentioned substances would cause the wax to sink; while sophistication with paraffin, ceresin, ozocerite, etc., would make it float upon the surface. Generally speaking, the principal adulterations of wax are with yellow ceresin for yellow wax and white ceresin for white wax. *Paraffin*, as well as *ceresin*, etc., is recognized by heating a sample of the suspected wax with fuming sulphuric acid in a porcelain dish. Pure wax is thereby entirely decomposed and converted into a black jelly-like mass, from which the paraffin is separated in an unchanged form after cooling. *Tallow*.—By pouring spirit of sal ammoniac over scraped wax in a test-tube, and heating, the fluid does not become turbid, as is the case in the presence of tallow. *Stearic acid* is detected by dissolving a small quantity of wax in ten times the quantity of chloroform and adding lime-water. Pure wax remains dissolved, while in the presence of stearin a granular precipitate of stearate of lime (lime soap) is formed. *Resin*.—Boil the wax with concentrated nitric acid;

in the presence of resin the fluid acquires a reddish color. By adding water to the fluid after cooling, and freeing it from wax, it becomes turbid and the resin separates as a yellow, flaky precipitate, which dissolves with a reddish-brown color in caustic ammonia. *Japanese wax.*—Boil about equal parts of borax and wax with fifteen to twenty times the quantity of water; with pure yellow wax the milky, turbid mixture separates gradually into a clear yellowish fluid, and with pure white wax into a clear fluid, with the wax in both cases floating on top. In the presence of Japanese wax the whole remains milky, and, according to the quantity of adulteration, thick or jelly-like and rigid.

The fusing point of yellow wax = 143.6° to 144.5° F., and that of white wax = 145.4° to 149° F., is lowered by an addition of paraffin, ceresin, tallow and Japanese wax, and raised by the addition of stearic acid, carnauba wax and resin; mixtures with the same fusing point may, however, be prepared.

Hehner has attempted to apply to the examination of wax the well-known method of Koettsdorfer for fats. The method of analysis is as follows: First make up alcoholic potash, using spirit rectified over potash of such strength that 1 cubic centimeter equals 0.3 to 0.4 cubic centimeter normal acid. Weigh 3 to 5 grammes of wax, transfer to a flask holding about 400 cubic centimeters, and heat on the water-bath with 50 c.c. alcohol prepared as above. Add plenty of phenolphthalein solution and titrate with the alcoholic potash. Next run in an excess of the alkali, 50 c.c. being a convenient quantity. The whole is then briskly boiled under a reflux condenser for an hour, shaking occasionally. The solution should be clear, or nearly so. Titrate back the excess of alkali with standard acid, keeping the liquid boiling. From the data thus obtained, the free acid calculated as cerotic acid and the saponifiable substance—calculated as myricin—are obtained. 1 cubic centimeter alcoholic potash = 0.41 gramme cerotic acid, or 0.676 gramme myricin. Tests have shown an average of 14.4 per cent. cerotic acid—molecular weight = 410—and 88.09 per cent.

myricin. In case of adulteration the proportion between free cerotic acid and myricin is changed, because the acids used for adulteration have a considerably less molecular weight; thus 284 parts stearic acid correspond to 410 parts cerotic acid. Hence there has to be calculated for

1 per cent. stearic acid = 1.443 per cent. cerotic acid.
1 " palmitic acid = 1.601 " "
Both acids in mixture = 1.510 " "

For 1 per cent. rosin—molecular weight = 329—Hehner calculates 1.246 per cent. cerotic acid. For 1 part of fat added 2.391 parts of myricin are calculated.

### *Coccina* (*Hemiptera*).

*Chinese wax* or *Pela wax* (*Insectenwachs*, *Chinesisches wachs*, G.; *cire d'insectes*, *cire d'arbre*, F.) is produced by *Coccus ceriferus*, Fabr. = *Coccus pela*, Westwood, or the white wax insect of China upon the branches of *Fraxinus chinensis*, Roxb., or the Chinese ash, and perhaps upon those of other *Oleaceæ*. The Chinese have found that the rearing of the wax insect and the production of wax are two different processes which cannot be advantageously carried on together, but may be brought to great perfection by suitable separation. The regions about Kien-tschang and Kai-ting-fu divide the labor. In Kien-tschang the ash is cultivated upon which the wax insect lives and increases, but separates very little wax. It is evidently there under conditions best adapted for its healthy development.

Toward the end of April the inhabitants of Kien-tschang leave their homes in great numbers, each with a bag of precious eggs upon his back and travel over steep mountain paths to Kia-ting-fu, which they reach in about fourteen days. The journey is chiefly made at night to avoid the heat of the day, as otherwise the sun might breed out the eggs too soon. In Kai-ting-fu six or seven of the bags of eggs are enveloped in a palm leaf and at once hung upon the young shoots of the ash. No further work is required. In a few days the insects begin to make their appearance and spread out like a brownish coating over the branches, without, however, touching the leaves. Gradually, as the insects grow, the surface of the branches becomes incrusted with a white substance—the wax—which is formed by direct secretion from the body of the female and the young insects, which remain imbedded in the wax crust.

In the latter part of August the branches are cut off and boiled in water, whereby the wax collects upon the surface. Later on it is remelted and poured into deep pans, where it cools to a transparent and very crystalline substance.

In Kia-tung-fu the insect lays no eggs, the climate, according to the opinion of the inhabitants, not being warm enough. The abundant secretion of wax seems to indicate a morbid condition produced by too luxurious food. Every attempt to produce good wax in other localities has failed.

Chinese wax or insect wax differs entirely from Japanese wax, with which it is frequently confounded, especially in literature. It is pure white or of a very slightly yellowish shade, tasteless and inodorous, lustrous and crystalline thoughout. In appearance it resembles spermaceti, but is much harder and more brittle, can be almost pulverized, and is of a more fibrous structure. It has a specific gravity of 0.970 at 59° F., melts at 179.5° to 181.5° F., yields little to alcohol and ether, but is very readily soluble in benzine. It is difficult to saponify with boiling potash lye.

Chinese wax is not a mixture of various substances, but consists almost entirely of ceryl cerotate $\left.\begin{matrix} C_{27}H_{53}O \\ C_{27}H_{55} \end{matrix}\right\} O$.

In China and Japan it forms an important article of commerce, being used in the manufacture of candles, which, it is claimed, possess ten times the illuminating power of ordinary candles. It is also used for coating candles manufactured from Chinese vegetable tallow (see Vol. 2, page 80); further, for polishing articles of leather and clay, and medicinally for external and internal use.

At the present time Chinese wax is not of much commercial importance, as China consumes immense quantities, but formerly it was brought to England in broad cakes perforated in the centre, and was much used in the manufacture of candles.

### *Cetacea* (*Mammalia*).

*Spermaceti* (*cetaceum; Walrat*, G.; *cétine, ambre blanc*, F.) is found in a state of solution in special cavities of the skull bones of *Physeter macrocephalus*, Linn., the sperm whale or the cachelot (see Vol. 2, page 50) and other whales, but after the death of the animal forms a crystalline mass. The whale oil is strained off, the remaining crystalline mass pressed and treated with a weak solution of caustic soda or caustic potash to saponify any adhering oil. It is then rinsed off in water and remelted in boiling water.

Crude spermaceti, which is seldom found in commerce, forms finger-thick, yellowish, transparent plates, of a foliated structure, and a rancid, fishy odor.

The article, treated with dilute lye, etc., comes into commerce in white, semi-transparent, broadly foliated, crystalline pieces, talcky to the touch, tasteless and inodorous, and friable. It has a specific gravity of 0.943 at 59° F., fuses between 122° and 129° F., and, like wax, solidifies immediately below the melting point. The purified spermaceti remaining, after repeated treatment with hot alcohol, has its fusing point several degrees higher, and can be distilled at 680° F. without perceptible decomposition and alteration.

Spermaceti yields nothing to water and very little to cold alcohol, benzine, and petroleum ether. Forty parts of boiling alcohol of 0.830 specific gravity dissolve one part of spermaceti, the greater portion of which, however, recrystallizes on cooling. It is readily soluble in ether, chloroform, and carbon disulphide.

Spermaceti is a mixture of various fats, but not of glycerides. By recrystallizing it from spirit of wine cetylic palmitate $\left.\begin{matrix} C_{16}H_{31}O \\ C_{16}H_{33} \end{matrix}\right\} O$ is formed. It contains besides small quantities of other combinations difficult to saponify, which by decomposition yield stearic, myristic, and lauro-stearic acids and the alcohols corresponding to these acids.

Spermaceti is composed of:—

| | | |
|---|---|---|
| Carbon | 80.03 | per cent. |
| Hydrogen | 13.25 | " |
| Oxygen | 6.72 | " |
| | 100.00 | " |

Spermaceti is difficult to adulterate, as by every admixture it loses in a high degree its peculiar and very characteristic properties. Adulterations are readily recognized by greater hardness, absence of nacreous lustre, and the small foliated crystalline structure. Some *wax* is generally admixed, but it should not be in such a quantity as essentially to increase the specific gravity or melting point or to make the solution in ether milky. *Tallow* is detected by its odor in melting and by the acrolein odor diffused in extinguishing a wick saturated with it. Besides, spermaceti adulterated with tallow makes fat stains upon paper, which is not the case with the pure article. An addition of *stearic acid* renders the spermaceti harder and smaller foliated, and, on boiling the suspected sample in soda solution, causes effervesence. *Paraffin* is detected with sulphuric acid in the same manner as in wax.

By continued exposure to the air spermaceti becomes yellowish and rancid, but it can be again made fit for use by remelting and treating with dilute caustic lye. By boiling in dilute solution of sodium carbonate, spermaceti is scarcely attacked, in which it differs from stearin.

Spermaceti, generally mixed with wax, is used in the manufacture of candles which burn with a bright, inodorous flame. In medicine it was formerly employed internally in irritation and inflammation of the alimentary canal and of the bronchial membrane, but its principal use now is in the preparation of ointments.

The United States and England furnish most of the spermaceti. It is brought into commerce in barrels weighing 450 to 850 pounds each.

French spermaceti is the finest variety; it is brought into commerce in square cakes weighing from 33 to 35 pounds each. There are, however, two qualities, one dry and pure white, and the other more fatty and somewhat yellowish. English spermaceti comes in the form of conical masses of 275 pounds each, two in a barrel. The inferior quality of American spermaceti is gray-yellow and contains much oil.

## CHAPTER XVI.

### WASTE FATS.

UNDER waste fats we understand, on the one hand, fats gained as by-products, for instance, wool fat, bone fat, etc., and on the other hand, the regaining of used fats from the soap waters of cloth mills and dye houses, from tanning waste, etc., such as fuller's fat, glue fat, degras (fish oil, which has been used in making chamois leather), etc.

This industry, which is of comparatively recent date, does not only augment the national wealth, but is also of great importance from a sanitary point of view, as it prevents the drainage of enormous quantities of waste water into the public water-courses.

*Wool fat or suint* (*Wollschweissfett,* G.; *oesype,* F.)—The wool of sheep, goats, etc., contains besides the actual wool substance a considerable quantity of fatty and mineral bodies, among the latter potassium carbonate. It is well known that the herbs and grass upon which the sheep pasture withdraw large quantities of potassium from the soil, which, after passing through the animal organism, are separated simultaneously with the sweat through the skin and deposited in the wool as wool sweat or suint. The potassium thus separated has a saponifying effect upon a portion of the fat which is formed, so that considerable quantities of potash soaps, stearic and oleic acids, and probably also palmitic and some valerianic acid, are present in the suint. There are further found compound ethers of cholesterin, for instance, cholesteryl benzoate $\left.\begin{matrix}C_{26}H_{43}\\C_7H_5O\end{matrix}\right\}O$, cholesteryl stearate $\left.\begin{matrix}C_{26}H_{43}\\C_{18}H_{35}\end{matrix}\right\}O$, and also free cholesterin and isocholesterin $C_{26}H_{44}O$, a substance isomeric with cholesterin.

Less suint is found in coarse wool than in fine, the average being about 30 per cent. of the crude wool.

Air-dried varieties of wool contain:—

| | | | |
|---|---|---|---|
| Moisture | 20.12 p. c. | 17.52 p. c. | 14.23 p. c. |
| Wool fat | 6.93 " | 9.16 " | 8.54 " |
| Suint | 22.35 " | 20.69 " | 22.40 " |
| Dirt | 9.80 " | 13.62 " | 23.13 " |
| Pure wool | 40.80 " | 39.01 " | 31.70 " |
| | 100.00 " | 100.00 " | 100.00 " |

The so-called suint is to be considered a mixture of secreted substances with accidental exterior ones. By macerating crude wool in warm water for some time, a turbid, foaming fluid is formed which contains the constituents of the suint partly in a dissolved and partly in a suspended state. The dry substance of the watery extract consists of:

| | |
|---|---|
| Potassium sulphate | 2.50 per cent. |
| Potassium carbonate | 44.50 " |
| Potassium chloride | 3.00 " |
| Organic substances | 50.00 " |
| | 100.00 " |

The potash which can be obtained from 100 pounds of wool amounts to from 7 to 9 pounds.

From the fat wool exhausted with cold water the wool fat can be extracted by ether and qnantitatively determined.

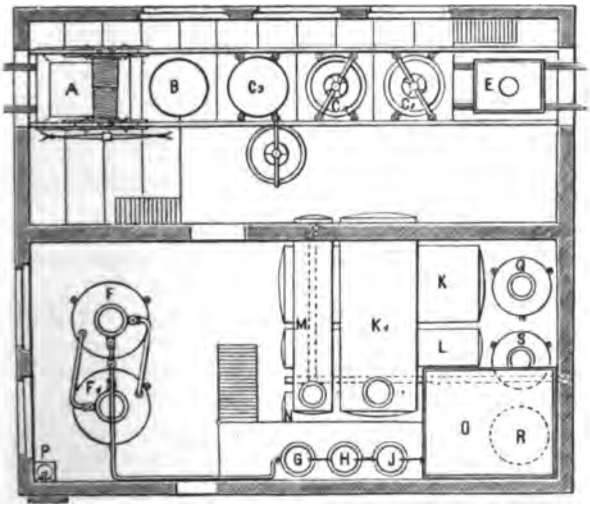
Before wool is brought into commerce it must first be converted into salable merchandise. This is effected by washing, shearing, and sorting.

The sorted wool, as found in commerce, contains always a considerable portion of suint, from which it has to be completely freed by washing to fit it for manufacturing purposes. This is done with the assistance of alkaline fluids, which must, however, be so weak as not to attack the wool fibre. The alkaline fluids generally used are a mixture of soft water and putrid human urine, or a solution of soap in soft water, or a weak solution of soda. The wash waters are either allowed to run off without being utilized, whereby the entire content of fat is lost, and the water of rivers and creeks contaminated to the injury of the public health, or the fat is regained by precipitation by means of iron, lime, etc., or, where local conditions permit, converted into illuminating gas.

The principal point with which we have here to deal is the extraction of the fat by means of a solvent. Carbon disulphide was first used, but it has the disadvantage of being difficult to regain without injury to the wool fibre. The use of dry, hot air for the removal of the carbon disulphide makes the wool hard, and steam felts it. The most suitable agent is moist, hot air. Richter, of Berlin, freed the wool from fat by means of amyl alcohol, but the loss of solvent was too large.

Wool can, however, be readily freed from fat by means of petroleum-ether and benzine, whereby it retains its softness and pliancy.

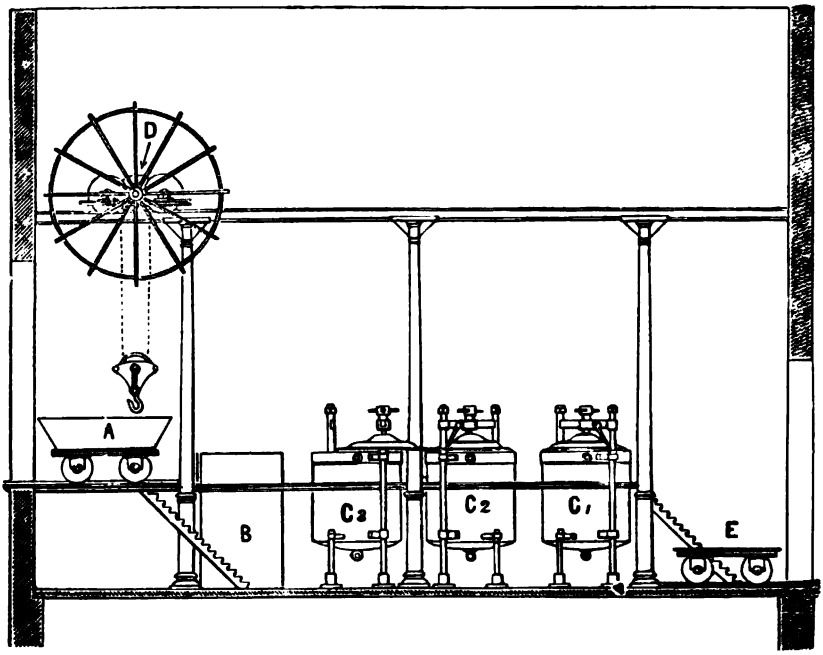
FIG. 236.



WOOL-EXTRACTING APPARATUS BY MEANS OF ETHER. GROUND-PLAN.

Figs. 236, 237, 238 and 239 illustrate Braun's process by means of ether, more fully described below. Its object is, that after a portion of the sweat and dirt is first removed by water, the latter is dislodged by alcohol, the alcohol by ether, the fat by more ether, the ether by alcohol, and the alcohol again by water. Or, in other words, water is allowed to stream through

FIG. 237.



WOOL-EXTRACTING APPARATUS BY MEANS OF ETHER. LONGITUDINAL SECTION THROUGH THE EXTRACTING ROOM.

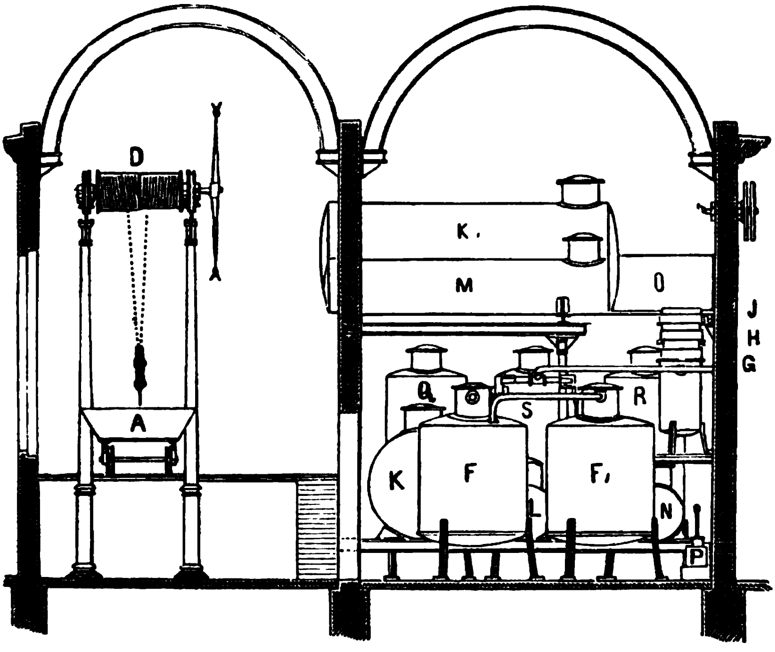
*A*, carriage; *B*, press cylinder; $C_1 C_2 C_3$, extracting vessels; *D*, differential windlass; *E*, carriage; *F F*, stills; *G H J*, cooling apparatuses; *K K*, ether reservoir; *L M*, alcohol reservoir; *N*, reservoir for mixed liquids; *O*, water reservoir; *P*, air-pump; *Q R S*, air-chambers.

the wool, then alcohol, then ether, again alcohol, and finally again water.

Of course, not only the fat is regained from the ethereal solution, but also the alcohol and ether used. The results obtained by the process are very satisfactory and the loss of materials used very small. The apparatus is also well adapted for extracting fat from waste wool, rags, leather, bones, etc.

*A* is a carriage upon which the wool to be extracted is carried into the factory, and *B* a cylinder of sheet or cast iron open at the top. It is one-and-a-half times as high as the vessels $C_1C_2$ and $C_3$, with which it is connected in a straight line and thoroughly secured. $C_1C_2C_3$ are the vessels in which the wool is extracted. They are cylindrical in form, of sheet or cast iron, have a curved bottom and an air-tight lid which can be readily removed. *D* is a transportable differential windlass. It serves to press together the fatty wool in the press cylinder *B* to about one-third its volume, to lift the package of compressed wool from the cylinder *B*, to transfer it to the extracting vessels $C_1C_2C_3$, and remove it after the extraction of the fat and place it upon the carriage *E*, which takes it from the factory.

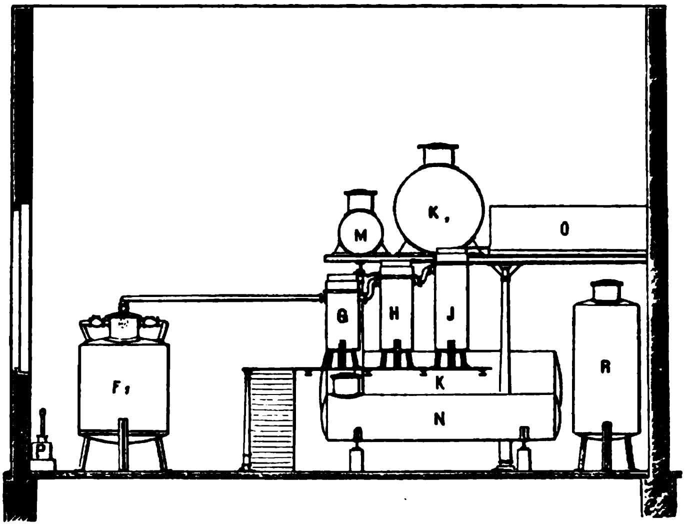
FIG. 238.



WOOL-EXTRACTING APPARATUS BY MEANS OF ETHER. CROSS SECTION.

*F* and $F_1$ are two cylindrical stills provided with double bottoms and a steam coil. *G* is a cooling apparatus of peculiar construction, which, during distillation, is kept at 176° to 212° F. to prevent the condensation of any considerable quantity of ether or alcohol. *H* is a cooling apparatus similar to *G*. It is kept at 122° to 158° F., so that no ether vapor can condense in it. *J* is an apparatus like *G* and *H*, and is kept as cool as possible by means of cold water. *K*, the reservoir for ether, is a cylinder of sheet iron and is placed upon the floor. $K_1$ is also a reservoir for ether like *K*, but is placed about ten feet higher than *K*. *L* is a reservoir for alcohol placed upon the floor. *M*, a reservoir like *L*, stands about ten feet higher than *L*. *N* is a reservoir like *L*, for mixed liquids, and rests upon the floor. *O* is a water reservoir placed higher than the

FIG. 239.



WOOL-EXTRACTING APPARATUS BY MEANS OF ETHER. LONGITUDINAL SECTION THROUGH THE DISTILLING ROOM.

**A**, carriage; **B**, press cylinder; $C_1C_2C_3$, extracting vessels; **D**, differential windlass; **E**, carriage; **F** $F_1$, stills; **G H J**, cooling apparatuses; **K** $K_1$, ether reservoirs; **L M**, alcohol reservoir; **N**, reservoir for mixed liquids; **O**, water reservoir; **P**, air-pump; **Q R S**, air-chambers.

cooling apparatus *J*. *P* is a double-acting air-pump. *Q* is a chamber for compressed air. *R* is a chamber for rarefied air. *S* is an air-chamber in which a constant atmospheric pressure prevails, it being connected with *Q* and *R* by self-acting valves.

All the vessels are connected by pipes, cocks, and valves, which are, however, not shown, to prevent the illustrations from being too complicated.

In the commencement of the work the reservoir *K* is filled with ether, *L* with alcohol, and *O* with cold water. The air-pump is then put in motion, so that the air in *R* is rarefied and in *Q* compressed. By then connecting $K_1$ and *M* with *R* and *K*, and *L* with *Q*, and opening the corresponding discharge-cocks on *K* and *L*, the ether ascends from *K* to $K_1$ and the alcohol from *L* to *M*.

During these preparatory operations other workmen fill the press cylinder *B* with wool to be extracted, and compress it between two sieve bottoms to two-thirds its volume with the assistance of the windlass. The package of wool is then lifted from *B* and placed in the extracting vessel $C_1$. The other extracting vessels $C_2$ and $C_3$ are charged in the same manner with wool and hermetically closed. Ether is then discharged from the reservoir *K* into $C_1$, first from below until all the air is expelled, and then from above. The ether forces the specifically heavier fat solution from $C_1$ to $C_2$, entering the latter first from below until all the air is also expelled, and then from above. The vessel $C_3$ is then connected with $C_2$ in the same manner as above described for $C_2$ with $C_1$.

After the extracting vessel $C_3$ is also filled with ethereal fat solution, the latter is brought into the still *F*. This fat solution is concentrated by having passed through the three vessels *C*.

When the wool in *C* is freed from fat, which is recognized by the evaporation of a few drops of ether taken from a test-cock on a pipe between $C_1$ and $C_2$, the flow of ether from the reservoir $K_1$ to the extracting vessel $C_1$ is stopped, and alcohol admitted from *M* to $C_1$ from below.

The specifically heavier alcohol drives the ether forward without mixing much with it.

When $C_1$ is about half filled with alcohol, which is recognized by a test-cock in the centre of $C_1$, the flow of alcohol from $M$ to $C_1$ is stopped, and water admitted from $O$ into $C_1$ from below.

The specifically heavier water drives forward the alcohol, and the latter the ether without mixing with it to any extent.

In a short time, before all the ether is forced from $C_1$ to $C_2$, the cock from $C_1$ to $C_2$ is closed, so that no alcohol and water pass into $C_2$, and the cock from $C_1$ to the still $F$ is opened, so that water and alcohol with some ether run direct from $C_1$ to $F$. The flow of water through $C_1$ is only continued until all the ether and alcohol are expelled.

When this point is reached the cocks on $C_1$ are closed, the lid is opened, and the wet wool taken out and placed upon the carriage $E$, by which it is carried into the rinsing-room. Simple rinsing removes all dirt, but to give the wool a brilliant lustre a weak soap-bath is required.

The extracting vessel $C_1$ is immediately refilled and the flow of liquids exactly regulated as before, except that the succession of the vessels is not $C_1$, $C_2$, $C_3$, but $C_2$, $C_3$, $C_1$.

After $C_2$ is emptied and refilled, the succession is $C_3$, $C_1$, $C_2$, then again $C_1$, $C_2$, $C_3$, and so on.

The fluids in $F$ are subjected to distillation. To save steam and cooling water two stills, $F$ and $F_1$, are provided. They are connected with each other in such a manner that the vapors from $F$ pass into the fluids in $F_1$, or the vapors from $F_1$ into the fluids in $F$.

From $F_1$, or $F$, the vapors consisting of vapors of water, alcohol, and ether pass into the cooling apparatus $G$, which is kept at a temperature of about 176° F., so that only water (with very little alcohol) condenses and flows back to $F$ or $F_1$. The vapors of ether and alcohol (with very little steam pass on to the cooling apparatus $H$, which is kept at a temperature of about 122° F. Here the alcoholic vapor and the steam still present condense, while the ether passes on to the cooling apparatus *J*, where it condenses in consequence of the low temperature (about 50° F.), which prevails therein.

The condensed cold ether runs back from the cooling apparatus *J* to the reservoir *K*, to be again used. The warm alcohol condensed in *H* runs through a coil placed in cold water to the reservoir *L*, and is again used.

Although it is not absolutely necessary that the ether condensed in *J* should be entirely free from alcohol, as alcoholic ether also dissolves the fat, it is best to make the separation as complete as possible. The air-chamber *S* is provided for this purpose. The condensation of vapors does not only depend on the temperature but also on the pressure under which they stand, and it is therefore not sufficient to keep the dephlegmating apparatuses at a certain temperature, but they must also retain the same pressure. The ordinary means of keeping the receiver open would cause considerable loss of ether. The air-chamber *S* is therefore connected with the cooling apparatuses in such a manner, by the self-acting valves *R* and *Q*, that the valve opens toward *R* when pressure is formed in *S* (or in the cooling apparatuses *J*, *H*, *G*), and remains open until atmospheric pressure is restored in *S*. By rarefaction in *S* (or in *J*, *H*, *G*) the valve opens towards *Q* until atmospheric pressure is restored in *S*.

The residue in the stills consists of fat and water, and is discharged into reservoirs. The fat can be used at once, as it is entirely pure.

The loss of ether, it is claimed, does not amount to 1 per cent.

It may be remarked that only wool from washed sheep can be used. Raw or sweat wool must first be soaked, and then freed from dirt by washing in warm water to which has been added some potash. The wool can be brought wet into the extracting vessels; in this case the water being first dislodged by alcohol and the latter by ether. The wash-water from sweat wool can be advantageously utilized for potash. The water is evaporated to dryness and heated upon the hearth of a reverberatory furnace. The residue contains the potash used in washing and the larger portion of the potassium of the wool in the form of potash.

The fat extracted from wool by means of ether, benzine or carbon disulphide has a yellowish to green-brown color and a characteristic wool odor. It is of a somewhat viscous and smeary consistency, and has a specific gravity of 0.973 at 59° F. It fuses at 102° to 104° F., and is sparingly soluble in alcohol. It is very difficult to saponify with alkalies, as it contains no glycerides, but cholesteral ethers of stearic acid $\left.\begin{matrix}C_{26}H_{43}\\C_{18}H_{35}O\end{matrix}\right\}O$, of oleic acid $\left.\begin{matrix}C_{26}H_{43}\\C_{18}H_{33}O\end{matrix}\right\}O$, and very probably palmitic acid, and besides valerianic acid, and a resinous substance.

From the behavior of wool-fat above described it will be readily understood that for a long time it was considered a troublesome by-product. The great amount of river pollution brought about by allowing the wash-waters to run into the nearest stream led to stringent decrees against this practice, and was primarily the cause of seeking some way of utilizing the actual fatty matters.

The first efforts were directed towards separating the fatty acids contained in the wool-fat and rendering them suitable for the manufacture of soap. Several methods for this purpose may here be mentioned, which in their principal features resemble one another.

For obtaining the fatty acids the wool-fat, according to Buisine, is heated with lime under pressure of 8 atmospheres, when the cholesterin is distilled off with superheated steam or the non-saponifiable admixtures are extracted by means of a solvent. From the lime soap remaining behind, the fatty acids are obtained by the addition of hydrochloric acid. In a similar manner the viscous slime obtained from the wash-waters by precipitation with lime may also be utilized.

Another method is as follows: For the preparation of fatty acids and oleic acids from wool-fat (German patent No. 32,015), the dried wool-fat is heated with dry solid alkali at a temperature of 482° to 572° F., the air being excluded. To exclude the air superheated steam or an indifferent gas is conducted through the apparatus.

By this reaction it is claimed the fatty acids in the wool-fat are first fixed without alteration as alkaline soaps, after which the alkali acts at a sufficiently high temperature upon the neutral ethers and decomposes them to their constituents, namely, oleic acid, stearic acid, cerotic acid, etc., which are fixed as soda soaps, and to alcohols, cerylic alcohol, etc.

These alcohols liberated by saponification are said to be converted by the alkali into corresponding acids, for instance, cerylic alcohol into cerotic acid, as the operation progresses and the temperature rises from 482° to 572° F. The soaps are lixiviated from the product of reaction by means of hot water from which they crystallize on cooling. They are then decomposed by acid, either directly or after conversion of the lime-soap. The resulting product is a mixture of fatty acids and oleic acid, which are separated by fractional distillation and pressing.

Both these methods have not been introduced to any extent into practice, the chief reason for this being very likely that they are too circumstantial to insure pecuniary success.

For the preparation of a saponifiable product of a light color from dark-colored wool fat, the latter, according to German patent No. 56,868, is converted by means of a boiling soda solution into an emulsion, diluted with a large quantity of water, and allowed to settle. The non-saponifiable cholesterins collect on the surface and are removed, while the remaining emulsion is decomposed by acid.

An emulsion of wool-fat was described by Dioscorides in his Materia Medica, and known as *œsypum*. Notwithstanding its disagreeable odor, it was highly esteemed as a remedy, and used as a cosmetic by the Roman women. A more refined product has of late years been introduced by Prof. O. Liebreich under the name of *lanolin*.

To prepare this material, Braun and Liebreich subject the suds in which fleeces have been washed to centrifugal action, whereby the crude lanolin and the dirt are retained separately while the purified soap water flows away. The crude lanolin thus obtained is first kneaded with water, then heated with water, and the wool-fat skimmed off. This wool-fat is for some time kneaded with water, whereby it is converted into white ointment-like lanolin. From commercial wool-fat an emulsion is prepared with the assistance of water, carbonate of soda, caustic soda, soap, etc. From this emulsion lanolin is obtained in the manner above described.

Later on Braun and Liebreieh's method was improved (German patent No. 38,444) in so far that the emulsion prepared from the suds in which fleeces have been washed, or from commercial wool-fat, are decomposed with dissolved salts of earthy alkalies (chiefly calcium chloride) to remove the dissolved soaps, the latter separating as insoluble combinations. The cholesterin fats are then separated by centrifugal action.

The crude lanolin is extracted with acetone, which dissolves the pure wool-fat, while the insoluble soaps as well as the dirt remain undissolved. It is best to use boiling acetone for extraction, whereby after cooling the solution or distilling off the acetone pure wool-fat free from acid is obtained, which by kneading with water yields lanolin.

*Or* the wool-fat obtained by centrifugal action is fused together with a small percentage of calcined marble, and the mass, completely freed from water, extracted with acetone.

Before treating the crude fat with lime salts or calcined marble, the coloring matter and odor are removed by means of oxydizing agents. The fatty acids are then saponified with aqueous alkalies and the assistance of heat, whereby they are converted into an emulsion resembling the suds in which fleeces have been washed. This emulsion is called "Wollfettmilch" (wool-fat milk) by the factory owning the patent.

The cholesterin fat extracted by means of acetone and freed from odor and color by oxidizing agents forms, after kneading with water, the commercial lanolin.

*Properties of lanolin.* Lanolin is a neutral and extremely pure wool-fat of a yellowish-white color and the consistency of a stiff ointment; it does not become rancid. The commercial product is an intimate combination of pure neutral wool-fat with about 30 per cent. water. Its most valuable property is the ease with which it takes up water and its rapid and ready resorbing power.

The most essential point in the manufacture of lanolin is the complete removal of the free fatty acids. A good quality of lanolin fit for every purpose should not show a greater acid reaction than freshly rendered lard.

The absorbing power for water of a few vehicles for salves as compared with lanolin is as follows:

| | | |
|---|---|---|
| 100 parts vaselin absorb | 4 | parts water. |
| 100 parts lard absorb | 15 | " " |
| 100 parts of a mixture of 70 parts almond oil and yellow wax absorb | 23 | " " |
| 100 parts of the same mixture with white wax absorb | 31 | " " |
| 100 parts of lanolin absorb | 105 | " " |

The quality and purity of lanolin are determined according to the following reactions:

Blue litmus paper rubbed with lanolin must not be reddened. The following reaction is more exact: Dissolve a small quantity of lanolin in benzine free from acid and add a drop of phenolphthalein solution; the addition of one drop of normal potash solution should produce reddening. After heating a short time with distilled water a clear pale-yellow (not brown) layer of oil and a clear aqueous layer should form. The latter must not change litmus paper. The separated layer of fat should dissolve clear in ether, and the lanolin leave no residue when burned upon a platinum sheet.

*Sulphuretted lanolin,* brought into commerce under the name of *thilanin,* is prepared, according to German patent No. 56,491, by heating lanolin with 20 per cent. flowers of sulphur to 248° F., whereby the greater portion of the sulphur is dissolved. The mass poured off from the undissolved portion of the sulphur is again heated to 446° F., whereby, under browning and the evolution of sulphuretted hydrogen, sulphuretted wool-fat is formed. Like wool-fat, it can be combined with water to a durable mixture—*sulphuretted lanolin.*

*Yorkshire fat.* The soap suds and similar waste liquors, resulting from the large consumption of soap and oil in the woolen industry, are treated in Yorkshire and other districts with acid, and the regained fat is used in the soap industry.

The soap suds and similar waste liquors are collected in large tanks or reservoirs holding 6000 to 8000 gallons, and acidulated with crude sulphuric acid (brown oil of vitriol) or hydrochloric acid. After agitation and subsequent standing for some hours, a fatty mass—the so called magma—deposits at the bottom of the tank. The supernatant aqueous fluid is then run off, and the tank filled up with fresh suds and acidulated as before, excepting that somewhat less acid is now requisite, owing to the smaller quantity of suds treated, the tank having been partly filled with magma and aqueous fluid. The magma is finally thrown on filter beds, where most of the remaining watery liquor separates, and then subjected to pressure in bagging. At first a very gentle pressure is applied, so as to squeeze out water only, but subsequently it is increased and heat applied, so as to filter the fused mass through the bagging. The residual press-cake is used as manure.

The product obtained by pressure is the so-called *Yorkshire fat*, a sticky grease of a brown to black color, and is sometimes used in the manufacture of soap, for oiling wool as well as for lubricating purposes, but more frequently it is subjected to distillation.

Distillation is effected in cast-iron stills resembling tar stills, and holding about 1000 gallons, or about 4 tons of fat. During the first 10 to 16 hours water is driven off with a direct fire, after which distillation is carried on with superheated steam.

Some fats, but not all, yield first some "*spirit-oil*," after which the principal product, the "*first distilled grease*," passes over, the latter being a thin, clear, pale yellow oil, which on cooling solidifies to a pale yellow fat. After the first distilled grease, which passes over in 20 to 24 hours, follows the "*green oil*," a greenish product, which is caught by itself and put back into the still, and worked over along with the next batch, or is occasionally used for coarse lubricating purposes.

About 36 hours after the commencement of distillation a thick oil comes over, when the operation is regarded as finished; the fires are then drawn, the superheated steam turned off, and the pitch remaining in the still run off.

This first distillation yields:

| | | |
|---|---|---|
| Water and loss | 20.76 | per cent. |
| Spirit oil | 4.10 | " |
| First distilled grease | 45.54 | " |
| Green oil | 15.47 | " |
| Pitch | 14.13 | " |
| | 100.00 | " |

To obtain a purer product the "first distilled grease" is frequently distilled a second time, so as to obtain about 96 per cent. of "*second distilled grease*" and 4 per cent. of "*soft pitch.*" The grease is then allowed to cool slowly, the temperature being for a few hours maintained at 70° F. By this means the stearin separates in a granular form, which facilitates pressing. Careful cooling of the fat is requisite so as to obtain the largest possible yield of olein with the use of the fewest number of press-cloths.

Granulation or "seeding" is occasionally effected in shallow, slightly-inclined metallic troughs. The olein exuding spontaneously from the crystallized cake is sometimes collected apart and designated *olein No. I.* It is claimed to be of better quality than that obtained by pressure.

The pressure under the hydraulic press of the fat wrapped in bagging is effected in six hours and yields about 66 per cent. of *olein* (*No. II*), which is clarified by filtering, and 34 per cent. of solid stearin.

By saponification with soda the stearin yields a soap which with water forms an extraordinarily gelatinous mass, from which free oil can be extracted only with difficulty by means of ether.

To the stearin 25 per cent. of paraffin may be added without mineral oil separating during saponification. This makes wool-fat stearin so valuable for the preparation of dressings, which mostly are mixtures of paraffin and stearin. On the other hand, soap prepared from the stearin is of little value, it dissolving with difficulty in water and yielding little lather.

The reason for the soap gelatinizing with water, as well as the peculiarity of the distilled fat of intimately mixing with mineral oil, has to be sought for in the content of non-saponifiable products formed during distillation, it being very difficult to conduct the latter in such a manner as to prevent the formation of such products. (See also "Fuller's Grease" later on.)

For the sake of completeness, two more methods also intended for the utilization of wool-fat, may be mentioned.

According to German patent No. 48,803 granted to the Norddeutschen Wollkämmerei und Kammgarnspinnerei at Bremen, *pure neutral wool-fat* is prepared as follows: The fatty mass separated by means of sulphuric acid from the soap suds, etc., is in a wet or dry state mixed with a solvent, such as benzine, carbon disulphide, etc., and solution of common salt, magnesium chloride, sodium sulphate, or another salt that withdraws water is immediately added. By this means the particles of soap separate from the solution of wool-fat, and the latter can be readily obtained by distilling off the solvent.

According to a second patent (No. 52,978) granted to the same firm, an extractive or gum resin, soluble in water, dilute alcohol and wool-fat, but insoluble in benzine, ether, chloroform, carbon disulphide, acetone and concentrated alcohol, is obtained from wool-fat. The fatty mass precipitated from the soap suds, etc., with sulphuric acid is treated, either before or after saponification, with benzine, ether, chloroform, carbon disulphide or other solvent, as well as alcohols, and the resulting solution purified with water or dilute alcohol, whereby this new product is claimed to settle at the bottom.

Another property of wool-fat that has thus far not been technically utilized is as follows: Rohart has found that wool-fat is capable of absorbing large quantities of sulphuretted hydrogen and combining with them. After saturation with sulphuretted hydrogen, the fat readily saponifies with soda at low temperatures, carbonic acid being evolved. Saponification may also be effected directly with alkaline sulphides without the evolution of sulphuretted hydrogen.

By this process, according to Borchers, a product is obtained which deviates completely from the properties of its constituents, and may be considered a soap. The sulphur soap thus obtained yields but little lather and has a slight, disagreeable odor. It might be useful as a remedial agent in skin diseases, otherwise it has no advantage over other soap.

*Bone fat* (*Knochenfett*, G.; *suif d' os, graisse d' os*, F.) The fat contained in the bones of animals is rarely obtained by direct boiling of fresh bones, but, as a rule, the bones are collected from slaughter-houses, hotels, kitchens, etc., and brought into storehouses, where they are sorted according to the purpose for which they are to be used.

Before use all bones must, however, be freed from fat. This is generally effected by simple boiling with water in open vessels, whereby, however, the bones are not completely freed from fat, only about one-third being obtained; or the bones are steamed as required for the manufacture of animal charcoal and bone meal. This so-called steaming is effected in upright cylinders with false bottoms. A jet of steam of a pressure of about 3½ to 6 atmospheres is introduced from below and allowed to act upon the bones for several hours. The fat obtained by either method is generally purified by remelting with common salt, that obtained from quite fresh bones being used used as axle-grease and that from old bones in the manufacture of soap.

Most factories working bones for any purpose have now introduced the extracting process for freeing bones from fat. There are a number of such apparatuses in use. The principle is the same in all, but for the sake of obtaining a patent some work with high pressure, some with low pressure, some with high and low pressure, etc.

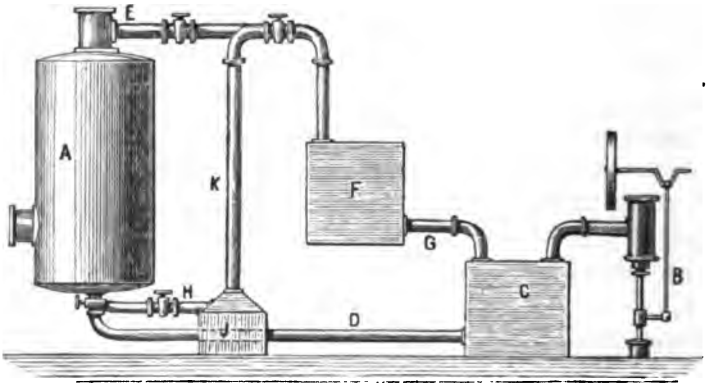
The old method of extracting the bones in a cold way, similar to rape seed, palm-nut kernels, etc., by means of carbon disulphide, did not free them entirely from fat, and required too much of the solvent.

Besides freeing the bones entirely from fat, the present method has the further advantage of improving the quality of the glue, animal charcoal, and bone meal.

In the following we give descriptions of some of the apparatuses.

*F. Seltsam's apparatus*, Fig. 240.—The bones are freed from fat by means of high-pressure vapors of carbon disulphide, benzine, and similar substances.

FIG. 240.



SELTSAM'S BONE-EXTRACTING APPARATUS.

*A*, cylinder; *B*, pump; *C*, reservoir; *D*, pipe for conducting the solvent; *E*, pipe for carrying off the vapors; *F*, condenser; *G*, pipe connecting the condenser *F* and reservoir *C*; *H*, pipe connecting the cylinder *A* and distilling apparatus *J*; *K*, pipe between the distilling apparatus *J* and the condenser *F*.

The cylinder, *A*, constructed for a tension of 10 atmospheres, serves as generator of steam and extracting vessel. It is filled with bones and hermetically closed. The necessary quantity of solvent is then brought by means of the pump *B* from the reservoir *C* through the pipe *D* into the cylinder *A*, and the latter heated. The formed vapors force the air through the pipe *E* into the condenser *F*, in which the vapors which may pass over are condensed and pass through the pipe *G* back into the reservoir *C*.

When all the air has been expelled from the apparatus and the pores of the bones, the valve on the pipe *E* is closed. The cylinder *A* is then heated, so that a pressure of a few atmospheres prevails in it. The tense vapors now act energetically upon the bones, the dissolved fat collecting upon the bottom of the cylinder; the valve on the pipe *H* is then opened and the superheated fluid discharged under strong pressure into the distilling apparatus *J*, and the solvent is distilled off from the fat by means of steam. The vapors of the solvent pass through the pipe *K* into the condenser *F*, and from there back into the reservoir *C*.

As soon as the manometer on *A* indicates no pressure, the valve on the pipe *H* is closed and the cylinder *A* further heated, with the pipe *E* open, so that any solvent still adhering to the bones can escape into the condenser *F* for condensation.

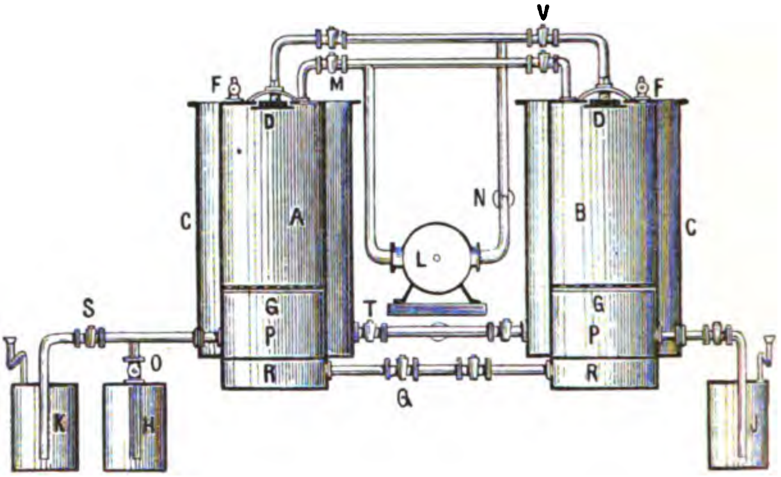
Figs. 241 and 242 illustrate Seltsam's apparatus as improved by Th. Richter, whereby the operation becomes entirely free from danger, the vaporization of the solvent being effected by steam only and the work carried on continuously.

There are two extracting vessels, *A* and *B*, of strong sheet iron and provided with double bottoms, the interspace serving for the reception of steam. The extracting vessels are surrounded by the jackets *C*. The extracting vessels are charged with bones through the man-hole *D*, and are further provided with the vacuum gauge *E* and the air-cock *F*. The bones are placed upon the perforated bottom *G* in the extracting vessels *A* and *B*.

There are besides two other vessels, *H* and *J*, which contain water, a vessel, *K*, for the solvent, and an air-pump, *L*. The operation is carried on as follows:

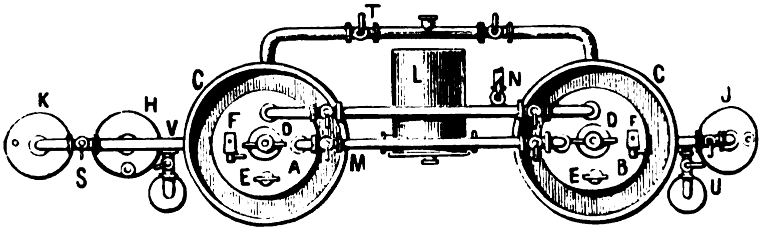
The extracting vessels *A* and *B* are charged with bones, all the cocks with the exception of *M* and *N* closed, and the air-

FIG. 241.



SECTION THROUGH THE APPARATUS.

FIG. 242.



GROUND PLAN.

*A B*, extracting vessels; *C*, jacket of *A* and *B*; *D*, manhole; *E*, vacuum gauge; *F*, air-cock; *G*, perforated bottom; *H J*, water vessels; *K*, vessel with solvent; *L*, air-pump; *M N*, cocks in the conduits to the air-pump; *O*, water-cock; *P*, space beneath the bottom *G*; *Q*, steam-cock; *R*, space between the double bottom; *S*, cock for the solvent; *T*, cock on the pipe between the extracting vessels *A* and *B*; *U*, discharge-cock for the fat; *V*, cock on pipe between the air-pump *L* and extracting vessel *B*.

pump *L* put in motion, whereby a vacuum is formed in *A*. When this is sufficiently large, water is admitted from *H* through the cock *O* into the space *P*. The water-cock is then closed and the steam-cock *Q* opened, whereby the steam entering the space *R* brings the water in *P* to the boiling point, and the air-pump sucks off the water vapors formed after the cock *N* is opened. The air-pump is then stopped and all the cocks closed except *S*, whereby the solvent passes from the vessel *K* into the space *P*, and, after closing the cock *S*, is evaporated by the admittance of steam into *R*. The water-cock is then opened and cold water admitted into the jacket *C*, which causes the solvent saturated with fat to condense in *P*. The water is then discharged from *C* and steam introduced into *R*, whereby the solvent is evaporated and forced into the extracting vessel *B* by means of the air-pump *L* after closing the cocks *M* and *V*.

The process is then repeated in *B*, after a vacuum has been created in the same manner as in *A*.

In the meanwhile the vacuum in *A* is interrupted by opening the air-cock *F*, and the separated fat is drawn off from *P* by opening the cock *U*.

The bones freed from fat are removed through the manhole *D*, and *A* is charged with fresh material while the process is carried on in *B*. Thus the operation is continuous, the solvent passing without any loss whatever from one extracting vessel into the other.

Th. Richter gives still another process for freeing bones, etc., from fat below the boiling point of water by low-pressure vapors of carbon disulphide, benzine, and similar solvents with low boiling points.

The substances to be freed from fat are brought into a suitable vessel, which can be hermetically closed. The vessel is then freed from air as much as possible by means of an air-pump and the solvent, for instance, benzine, admitted and absorbed by the bones. As soon as the latter are saturated the air-pump is put in motion, whereby the boiling point of the solvent is lowered and the entire space filled with its vapors. After allowing these vapors to act for some time, air is admitted, whereby the solvent is reconverted into a liquid state and dissolves the fat. The fat solution collects in the lower part of the vessel, after which the solvent is again vaporized. The vapors sucked off by the air-pump are condensed in a cooling apparatus. The application of this process becomes of special importance where either the substance to be freed from fat or the fat itself would suffer from the action of heat.

*Alfred Leuner's* process of freeing bones from fat by means of liquid vaporizable solvents and steam at a low pressure consists in first steaming the bones.

This is done in a boiler *A*, Fig. 243, provided with perforated bottom *B*. It is filled with bones and closed steam-tight. By steaming the air is expelled from the pores of the bones, the fat rendered more soluble, and the formation of glue induced. Steam is introduced through the pipe *C* and, after it has acted, the pressure is removed by opening the cock *D*. The condensed water is discharged through the cock *E*, and the cylinder *F* filled with an equal volume of water and benzine, or other solvent, the water forming the lower layer into which enter the pipes *G* and *H*. There is further a receiver *J* and a reflux cooler *K*.

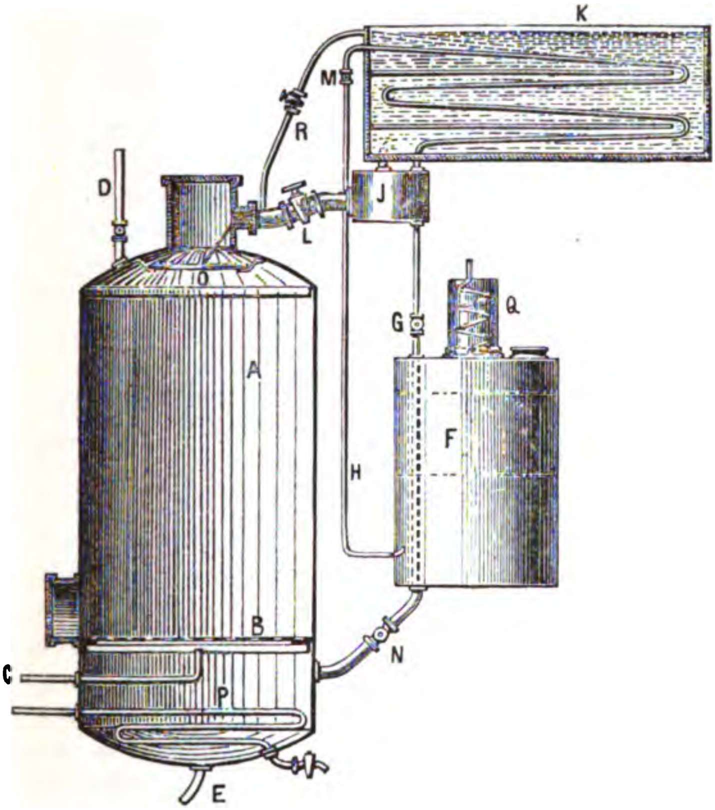
The bones are treated with vaporized solvent and with steam at an ordinary pressure, the steam essentially assisting the formation of benzine vapors and completing the formation of glue. By this process the decomposition of the nitrogenous substances of the bones in consequence of a high temperature is avoided; the products of bone meal being therefore richer in nitrogen and more valuable for manure.

The boiler *A*, receiver *J*, reflux condenser *K*, and vessel *F*, with the small reflux pipe *Q*, are connected by pipes which can be closed by cocks.

After steaming, the cocks *D* and *E* are closed and *L*, *M*, and *N* opened. The contents of *F* empty into the boiler *A*, where they are heated by steam passing through *P*.

The boiler *A* is consequently filled with vapors of benzine and water, which, after penetrating the bones, pass through the pipe *L* and the receiver *J* into the condenser *K*, where they are condensed; they then return in a liquid state to the receiver *J*. As soon as *J* is filled to the mouth of *L* the reflux commences through *L* to *A*, the fluids, after being converted into spray by the arrangement *O*, falling upon the bones.

FIG. 243.



LEUNER'S BONE-EXTRACTING APPARATUS.

*A*, boiler; *B*, perforated bottom; *C*, pipe for admitting steam; *D*, pipe for discharging steam; *E*, pipe for carrying off the condensed water: *F*, cylindrical vessel; *G*, cock on pipe between the receiver *J* and the cylindrical vessel *F*; *J*, receiver; *K*, reflux condenser; *L*, pipe and cock between the boiler *A* and receiver *J*; *M*, cock on pipe *H*; *N*, cock on pipe between the vessel *F* and boiler *A*; *O*, spray arrangement; *P*, steam coil; *Q*, small reflux condenser; *R*, pipe and cock between boiler *A* and reflux condenser *K*.

Vapors not completely condensed return through the pipe *G* to the vessel *F*, the last remnant condensing in the small reflux condenser *Q*. Benzine vapor and water vapor, as well as liquid benzine, act, therefore, for some time upon the bones. The cock *L* is then closed and, after opening the cock *R*, the contents of *A* are boiled for some time. The vapors pass into the condenser *K* and are condensed by means of a second coil indicated in the illustration by dots. The condensed benzine and water then run into the receiver *J*, and, after opening the cock *G*, into the vessel *F*, where the benzine collects.

This redistillation is carried on until water only is distilled, which is recognized by a sample taken from *J*. Water and fat are then discharged through the cock *E*, the fat is separated from the fluid, to be utilized in the manufacture of glue, and the bones are further worked.

*J. Wellstein and B. Birkenheuer's process.*—The extracting vessel *A* (Figs. 244 and 245), provided with steam-jacket, vacuum gauge, and manometer, is filled with comminuted bones through the opening *a*. The cock *b* on the steam-pipe *G* then is opened, so that the steam enters the condenser *B*, while the air contained in it escapes through the pipe *c*. The steam-cock and pipe *c* are then closed and cold water injected into the condenser by means of the pump *C* through the pipe *u* until a vacuum is formed. The cock *d* on the pipe *H* is then opened so that air is sucked into *B* from the boiler *A*, which is then expelled in the same manner by steam. The cocks *g* are then opened so that the solvent in the reservoir *D* passes through the pipe *K* and *h* into the boiler *A*, after which the cocks are again closed. The cock *P* is then opened and steam of a pressure of five atmospheres admitted into the steam-jacket, the air-cock *i* of which has been previously opened. As soon as there is a pressure of five atmospheres in the boiler *A* the steam is shut off, whereupon the vapors in the boiler condense and the fat in the bones separates and collects below the perforated bottom *k*. After the pressure in the boiler is reduced to 0.2 atmosphere, the solvent is allowed to flow back through the pipe *h* and *K* into the receiver *D*. To prevent any fat from being carried away the pipe *h* can be shifted by the wheel *l*, keyed upon it in such a manner that its lower end is screwed

Fig. 244.

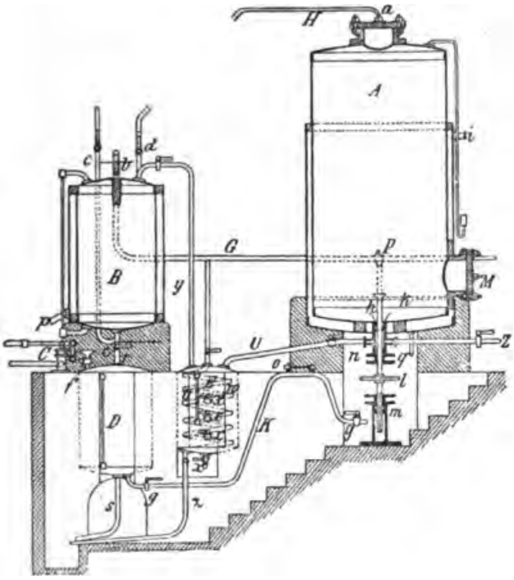
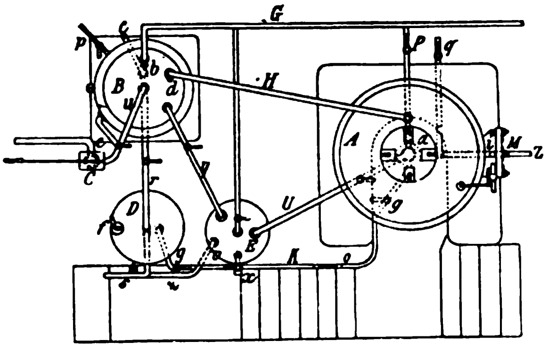


Fig. 245.



into the support *m*, which is provided with a stuffing box, and the upper end is guided by the stuffing box of the support *n*. In case any fat passes over, which is recognized by the glass tube *o*, the pipe *h* is screwed up by turning the wheel *l* until no more fat escapes, so that, it is claimed, all the solvent except a very small remnant can be carried off without gasification. The remnant of solvent, which is mostly contained in the bones themselves, is vaporized by steam introduced into the jacket of the boiler *A*. The vapors pass into the condenser, where they are liquefied by introducing cold water through the branch-pipe *e* into the jacket of the condenser *B*. The solvent then returns through the pipe *r* into the reservoir *D*, from which it can be discharged, together with the water passed over, through the pipe *s*. The cooling water runs off from the condenser through the pipe *p* and the condensed water from the jacket of the boiler *A* through the pipe *q*. The fat collected under the perforated bottom *k* is discharged through the pipe *U* into the fat reservoir *E*, which is provided with a stand-pipe *v*. The fat is purified by adding two per cent. of potash lye and introducing steam into the coil *w*, and then discharged through the cocks *x*. The fat discharged through the upper cock solidifies, it is claimed, at 39° to 43° F., and that through the lower cock at 59° to 68° F. The vapors developed thereby pass through the pipe *y* into the condenser *B*. The condensed water formed in the coil *w* runs off through the pipe *z*. In order to commence glue boiling immediately after the discharge of the fat from the boiler, steam is introduced into the latter until the bones are completely extracted. The glue solution formed is discharged through the pipe *Z*, and the bone residue removed through *M*.

*The American patents for extracting fats from bones by the agency of liquid hydrocarbon*, which are, we believe, owned by the firm of Baeder, Adamson & Co., of Philadelphia, are as follows:

A mode of extracting oily, fatty, and resinous matter from animal and vegetable substances by causing liquid hydrocarbon to trickle through a mass of such substances in a closed vessel, and preventing it from being subjected to steeping or flooding, is shown in Figs. 246 and 247. It is the invention of Messrs. Wm. Adamson and Charles F. A. Simonis, of Philadelphia, Pa., and relates to the treatment of animal and vegetable substances with hydrocarbons for the purpose of extracting therefrom oily, fatty, and resinous matter; and the object of this invention is to cause hydrocarbon to trickle through such substances instead of flooding the same, so that it will take up the oily, fatty, and resinous matter without any of the albuminous or gelatinous ingredients.

FIG. 246.

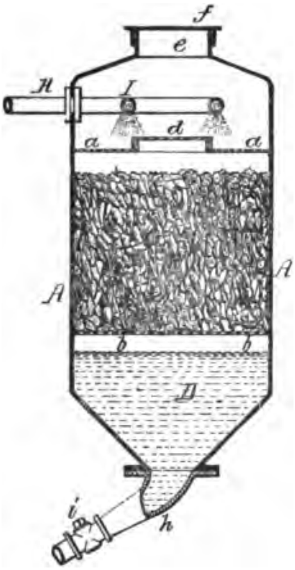


FIG. 247.

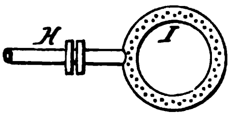


Fig. 246 is a vertical section of apparatus wherewith this invention may be carried into effect; Fig. 247, an inverted plan view of a part of Fig. 246.

*A* is a vessel, preferably of cylindrical form, and containing an upper perforated diaphragm *a*, and lower perforated diaphragm *b*, the former having a central opening, through which the material to be treated may be introduced between the two diaphragms, and this opening having a detachable perforated çover *d*.

On the top of the vessel there is an opening *e*, furnished with a detachable cover *f*, and at the bottom of the vessel there is an outlet-pipe *h*, furnished with a suitable cock or valve *i*.

Liquid hydrocarbon, preferably such as is of a volatile character—benzine, benzole, or gasoline, for instance—is introduced into the vessel above the diaphragm *a* through a pipe *H*, and perforated ring *I*, or otherwise, the hydrocarbon passing through the diaphragm and falling in a shower on to the substance contained in the vessel.

The hydrocarbon will trickle through the mass, taking up whatever oily, resinous, or fatty matter it comes in contact with until it falls through the lower diaphragm into the space *D*, whence it may be drawn off from time to time through the outlet-pipe *h*.

In extracting oily, fatty, or resinous matter from vegetable or animal substances by hydrocarbons, it has been the practice either to subject them to hydrocarbon vapors, or to immerse or steep the substances in hydrocarbon, until the latter takes up the oily, fatty, or resinous matter.

The vapor plan is preferable in treating wet animal substances, such as offal; but for dry vegetable or animal matter —seeds, for instance, or residuum resulting from the rendering of tallow—we prefer the process of trickling the hydrocarbon over the material, previously described.

The flooding or steeping of animal or vegetable matter in liquid hydrocarbon results in a mixture or emulsion of gelatinous, albuminous, and fatty or oily matter, combined with animal or vegetable tissues, the whole forming an amalgamated mass; hence, whatever fatty or oily matter is extracted is accompanied with more or less of the suspended gelatine or albumen, either of which is difficult to remove from the oil or fat, and has a tendency to discolor the same.

This difficulty, it has been found, can be obviated by preventing the hydrocarbon from remaining in a quiescent state in contact with the material; in other words, by causing it to trickle through the mass, which, by this plan, retains its granular condition, and gives out its oil or fat to the hydrocarbon without the albuminous or gelatinous matter.

In the apparatus before described, for instance, an occurrence of the objectionable flooding of the material, tending to bring about the results previously mentioned, is obviated by never permitting the extract in the lower portion of the vessel *A* to reach the lower diaphragm *b*. By drawing off the extract from time to time, any impediment to the free discharge of the hydrocarbon with such oily and resinous matter as it has taken up through the lower diaphragm is prevented, and a continuous dripping of the hydrocarbon through the mass secured.

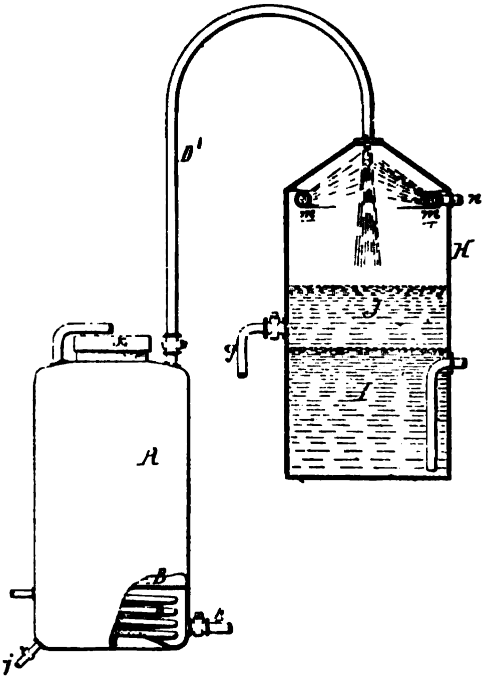
The extract obtained by the trickling or filtering process is much more concentrated than that obtained by the steeping and flooding process.

*Adamson's method for treating substances with hydrocarbon vapor, for the purpose of extracting oils, fats, etc.*—This improvement is intended to prevent the fetid or other odors imparted to the vapor from the substances treated from being recommunicated to the said substances, and to the extracts obtained therefrom through the medium of the vapor from the re-used hydrocarbon. The vapor is obtained from benzine, benzole, etc.

Fig. 248 represents, partly in section, the apparatus whereby the invention may be carried into effect.

*A* is a vessel in which the substances have to be treated by hydrocarbon vapor, the said substances being introduced into the vessel through a man-hole *x* and deposited on a perforated diaphragm *B*, the man-hole being provided with a suitable cover. A steam-coil *D* is placed in the vessel in a space beneath the diaphragm, and liquid hydrocarbon is introduced into the said space, and is there vaporized by the steam-coil. The vapor rising through the perforated diaphragm permeates the substance upon the same, so as to extract therefrom the oily, fatty, or resinous matter, which passes downward through the diaphragm into the space below the same, whence it may be drawn off from time to time through the discharge-pipe *j*. Liquid hydrocarbon may be introduced from a tank, or from a source explained hereafter, into the top of the vessel *A*, so that it will pass through the material and be vaporized when it reaches the coil; the said material being in this case subjected

FIG. 248.



to a downward current of liquid hydrocarbon and an upward current of vapor.

Previous to this invention it was Mr. Adamson's practice to cause the vapor, after acting upon the substances in the vessel, to pass through a worm in a condenser, the lower end of the worm communicating with the vessel *A*, beneath the diaphragm, as shown in Fig. 250, page 195, so that the hydrocarbon was used over and over again. But in practice this has been found objectionable in many cases for the following reason:

In treating animal offal, for instance, for the extraction of fats, fetid odors are imparted to the hydrocarbon vapor, and remain, to a considerable extent, in the condensed vapor when the latter is restored to the vessel *A;* hence the fetid odors were recommunicated both to the fatty extracts and to the material. The same objections have been experienced in treating meat for preservation, and vegetable matter for the extraction of oil by hydrocarbon vapor.

This difficulty is obviated in the following manner: The vapor-pipe *D'* communicates with a vessel *H* at the top of the same, and the vapor is met by numerous small jets of cold water—in the present instance from a perforated tubular ring *m*, into which the water is forced through a pipe *n*.

Many different appliances may be used, such as roses, revolving jets, etc., for causing a spray through which the vapor must pass, and by which it must be condensed. The result of this will be a supply, *I*, of tainted water on the bottom of the vessel *H*, and a quantity, *J*, of washed and purified hydrocarbon above the water, the latter having taken up the fetid odors.

The washed hydrocarbon may be drawn off through a pipe *g* into any suitable vessel, and thence introduced through the pipe *h* into the vessel *A*, or may pass directly into the latter to be again vaporized therein, the vapor, after permeating the material and passing through the pipe *D'*, being simultaneously condensed and washed in the vessel *H*, preparatory to being returned in the condition of purified liquid hydrocarbon to the vessel *A*.

By the practice of this process the inventor is enabled to obtain a purer extract than heretofore, and, at the same time, the substances acted upon are more free from noxious odors.

Changes may be made in the apparatus shown in Fig. 248, as, for instance, the vessel *A* may consist of a horizontal hollow cylinder, and the vaporizing of the hydrocarbon may be accomplished otherwise than by a steam-coil.

*Adamson's method for treating substances with liquid hydrocarbon for the purpose of extracting oils, fats, etc.*—This invention, which is that of Mr. Wm. Adamson, relates to a method of treating animal and vegetable substances with liquid hydrocarbons, such as benzine, benzole, etc., for the purpose of extracting from such substances oils, fats, etc.

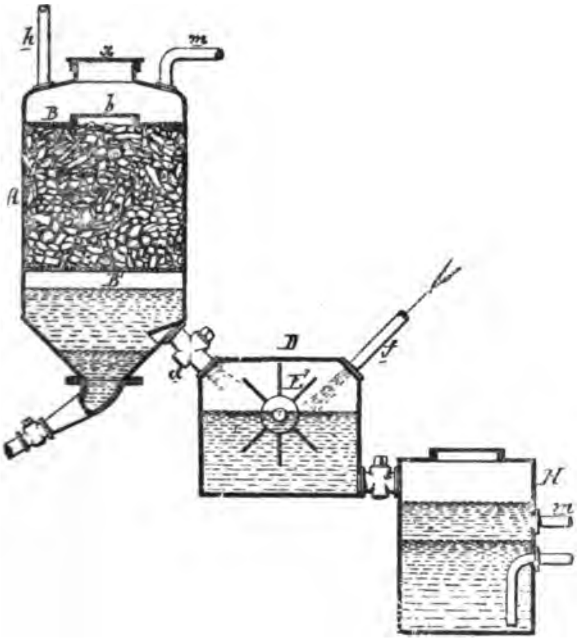
The object of this improvement is to prevent the fetid and other odors imparted to the liquid hydrocarbon by the substances treated from being recommunicated to substances and to the extracts therefrom by the liquid hydrocarbon when re-used.

In Fig. 249 there is shown a sectional view of apparatus whereby this invention may be carried into effect.

*A* is a vessel into which the substances to be treated are introduced through a manhole *x*, provided with a suitable detachable cover, and through an opening in the upper perforated diaphragm *B*, a detachable perforated plate *b* being placed over the opening after the substances have been passed through the same, the substances being supported by the lower perforated diaphragm *B'*, beneath which is a space for receiving the extract and liquid hydrocarbon after the latter has percolated through the mass in the vessel. The extract, which occupies the lowest position in the vessel, may be removed therefrom from time to time prior to being purified by distillation or otherwise. The liquid hydrocarbon is permitted to pass from time to time through a pipe *d* into a vessel *D*, where it is met by jets of water from a pipe *f*, the hydrocarbon and water being thoroughly agitated in the vessel by a revolving paddle-wheel *E*. This washing of the liquid hydrocarbon may be accomplished by different appliances. For instance, the paddle-wheel may be dispensed with, and water forced upward into the vessel from below in the form of numerous small jets. The water and hydrocarbon after this washing operation are permitted to pass into the subsiding vessel *H*, the hydrocarbon being above and the water below, the fetid and other odors divided by the hydrocarbon from the substances in the vessel *A* having, during the washing operation, been transferred to the water, which may be drawn off from time to time.

The washed and purified hydrocarbon may be pumped directly through a pipe *m* into the vessel *A*, to be re-used for treating the substances therein; or it may be pumped, first, into a reservoir, and permitted to flow from the same into the said vessel *A*.

FIG. 249.



More or less hydrocarbon is wasted by being drawn off with the extract, and to make up for this loss a supply may be introduced at intervals from a tank through the pipe *h*.

By the practice of the process described above, the inventor is enabled to obtain a purer extract than by the ordinary process of treating substances with liquid hydrocarbon. At the same time the substances treated will be much more free from noxious odors than when the hydrocarbon is used over and over again without washing.

It is not essential strictly to adhere to the apparatus shown in Fig. 249, as the construction of the apparatus will, in fact, depend in a great measure on the locality in which it is to be situated.

*Adamson's process for removing hydrocarbons from substances which have been treated therewith.*—This process, which is the invention of Mr. William Adamson, consists of washing from animal and vegetable substances the hydrocarbon which they retain after being treated therewith for the extraction of oils, fats, etc., and for other purposes.

Different apparatus may be employed for carrying out this process, and it may be conducted in the same vessel in which the material is treated with hydrocarbon.

The vessel which is shown in Fig. 250 has been found to answer well for this purpose.

This vessel is furnished with a suitable detachable cover *a*, and with two perforated or wire-gauze diaphragms *b* and *d*, both extending across the interior of the vessel, one near the top and the other near the bottom of the same.

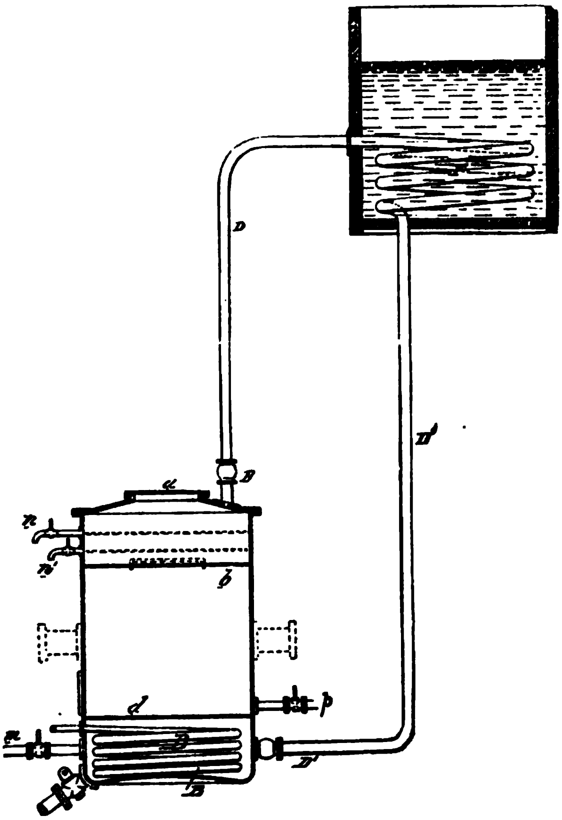
A steam-coil *B*, communicating with any adjacent steam-generator, is contained in the vessel below the lower diaphragm to vaporize the hydrocarbon, the vapor passing through the substance between the two diaphragms and out through the pipe *D*, which passes through a condenser *E*, the latter restoring the hydrocarbon to a liquid form, in which it is reconveyed to the vessel through a pipe *D′*.

In practising the washing process a pipe *m*, to introduce water into the vessel, and one or more outlet-pipes *n n′*, two in the present instance, are necessary. There may also be a pipe *p*, through which air can be introduced into the vessel under the circumstances explained hereafter.

When the treatment of the material in the vessel with hydrocarbon vapor, or liquid hydrocarbon, has been completed, steam is cut off from the coil *B*, the pipes *D* and *D'* are closed, and the cover *a* may be removed.

Water is now admitted through the pipe *m* to the space in the vessel below the diaphragm *d*, and the cocks of the outlet-pipes *n n'* are opened.

FIG. 250.



The water permeates the material, passes upward through the same, and carries with it the hydrocarbon, the latter having a tendency to rise with the water.

As the water, and whatever hydrocarbon accompanies it, pass through the upper diaphragm *b*, the hydrocarbon will at once rise to the surface, and will pass through the upper outlet-pipe *n* into any suitable receptacle, the water passing off through the lower outlet-pipe.

If this mode of separating the hydrocarbon from the water is practised, the admission of water to the vessel should be such in respect to the outflow that the liquid will remain at or near a uniform level; that is, the surface of the liquid should bear the relation shown in the drawing to the upper outlet.

The water and hydrocarbon, however, may be drawn off indiscriminately into a suitable receptacle, and then separated by decantation; but it is advisable in all cases that the water should extend above the mass of material in the vessel, so that the hydrocarbon can at once rise to the surface as it escapes from the substance.

When the material is of such a character as to be closely packed and not easily displaced by the upwardly-flowing water (and this is especially the case with seeds which have been treated with hydrocarbons), it is necessary to agitate the mass, so that the water can gain access to every part thereof. This agitation the inventor prefers to effect by air under pressure introduced through a pipe *p*, although mechanical appliances may be used for the purpose.

It will be understood that the process may be conducted in a vessel separate from which the substances have been treated with hydrocarbon. A vessel similar to that shown, for instance, but without the coil and pipes *D D'*, may be used, and may be furnished with trunnions (shown by dotted lines) and adapted to bearings, so as to be easily tilted when its contents have to be removed; or the vessel may have an opening near the lower diaphragm for the withdrawal of its contents, a suitable detachable door being adapted to the opening.

For the extraction of oil, especially from *bone fat* liquid, sulphurous acid is used according to the patent of W. Grillo and M. Schroeder (German patent No. 50,360). This method is based upon the observation that at 77° to 86° F., 3 parts of bone-fat yield, with 2 parts of sulphuric acid, a perfectly homogeneous fluid. The process is especially recommended for freeing bones from fat for the preparation of glue.

It is best to use an upright iron cylinder provided with the requisite apertures for introducing and discharging the bones, etc., and a perforated false bottom. Below the latter is a steam coil as well as the necessary apertures closed by cocks for introducing the liquid sulphurous acid and drawing off the coil. Since on account of the low boiling point of liquid sulphurous acid, the work has to be carried on under pressure, a valve for closing the cooling worm connected with the upper end of the apparatus is required.

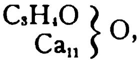
The apparatus being filled with bones and the apertures closed, the necessary quantity of sulphurous acid in a liquid state is brought under pressure into the apparatus, air containing acid being at the same time blown in through the upper valve of the cooling worm. The liquid sulphurous acid is then gradually heated by means of the steam-coil to from 86° to 104° F., whereby a tension of about 5 to 6 atmospheres is gradually developed in the apparatus. The acid in the form of gas ascends into the material to be freed from fat, partially liquefies therein, and, mixed with oil, runs back. When the heat of the matetial has increased to the above mentioned temperature, further condensation and the constant reflux of the sulphurous acid are effected by the worm cooled with water.

If coarse substances, for instance bones, are to be freed from fat, it is of advantage to interrupt heating from time to time, and by sucking off the gaseous sulphurous acid by means of a pump, to lower the pressure as well as the temperature in the apparatus. The effect of this is that the sulphurous acid mixed with oil contained in the bones is again partially gasified, and forces the oleiferous fluid from the pores of the bones. By now repeating the heating process, the gaseous acid in the interior of the material is again liquefied.

When the operation of freeing the material from fat is finished, the solution of oil in sulphurous acid at the bottom of the apparatus is subjected to distillation in the apparatus itself, or in a special still, in order to remove the sulphurous acid. For this purpose the cooling worm is used in a reverse direction, its lower end being connected with a closed boiler which serves as a receptacle for the liquid acid. For removing the acid gas remaining in the apparatus an air-pump has to be used, which forces the gases back into the reservoir. The last traces of acid are removed by blowing in air.

In freeing bones from fat the liquid sulphurous acid used can, after the operation is finished, be directly converted into watery solution, which may be employed for the lixiviation of phosphate of lime from the bones. In this case the use of a pump is not required.

---

Fat prepared by boiling fresh bones has a white to somewhat yellowish color, a slight odor and taste, is soft and ductile at ordinary temperatures, semi-fluid in summer, melts at 70° to 71.5° F., and solidifies at 59° F., the temperature being thereby increased to 62.6° F. It contains the glycerides of stearic, palmitic, and oleic acids. Exposed to the air, bone fat remains unchanged for a long time without becoming rancid, and, for this reason, is well adapted for lubricating machines. The fat obtained from old bones by steaming has generally a brown color and disagreeable odor, which is very repulsive if putrid bones have been used. The fat is somewhat granular, of a smeary consistency, and retains very much water. It fuses at 73.5° to 75° F. and solidifies at 62.5° F. It contains, besides stearin, palmitin, and olein, free fatty acids and two per cent. of calcium lactate  $\left.\begin{matrix}C_3H_4O\\Ca_{11}\end{matrix}\right\}O$, besides lime soaps, calcium stearate, calcium oleate, etc., which are soluble in the fat. The latter calcium combinations cause difficulties in saponification, and must therefore be decomposed in the melted fat by hydrochloric acid, and the fat purified by washing.

Extracted bone fat has a yellow to brown-yellow color and a very disagreeable odor, which is not removed by saponification.

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It is more solid than the other varieties, somewhat crummy, fuses at 79° to 80.5° F., and solidifies at 70°F.

The last two varieties of bone fat named are exclusively used in the manufacture of soap; however, never alone, but in combination with palm oil, tallow, etc.

Ten thousand hundredweight of bones yield, with the use of high or low pressure and a solvent, as much as 750 hundredweight of bone fat.

In the examination of commercial bone fats Valenta has established the following values:

1. *Content of water;* by drying a weighed quantity of the fat under examination at 230° F.

2. *Content of ash;* incinerate 5 to 7 grammes of fat in a platinum dish, weigh the ash, and determine the lime contained in it by itself.

3. *Determination of the total fatty acids;* this is effected according to the methods given, Vol. 1, p. 201.

4. *Determination of the free fatty acids;* this is effected in the most simple manner by boiling 5 or 6 grammes of fat in a small flask with the corresponding quantity of absolute alcohol, and after cooling titrating with decinormal potash to neutralization.

5. *Determination of the saponification value, of the iodine number and the fusing point of the fatty acids,* is effected according to the methods given, Vol. 1, p. 185.

The determination of the total fatty acids, and of the fusing points of the fatty and free fatty acids suffices in most cases for practical purposes.

| | |
|---|---|
| Fusing point of the fatty acids | 86° F. |
| Solidifying point of the fatty acids | 82.4° F. |
| Saponification number | 190.9 |
| Iodine number | 46.3 to 49.6 |

Iodine number of the fatty acids:

| | |
|---|---|
| Crude fat | 57.4 |
| Refined fat | 55.7 to 57.3 |

*Glue fat* (*Leimfett*, G.; *graisse de colle*, F.). The waste from slaughter-houses and tanneries, which is generally used in the manufacture of glue, must, before boiling, be freed from all fleshy and bloody particles, and especially from fat, which is effected by treating it with lime. By this operation the particles of flesh and blood are dissolved, while the fatty substances are saponified and form an insoluble lime soap which, by the subsequent boiling of the glue material, collects upon the surface and is skimmed off. This forms the so-called "glue fat" and contains from 40 to 50 per cent. of grease.

Crude glue fat contains fibres and other impurities, and on exposure to air constantly exhales ammonia, volatile fatty acids, and other disagreeable gases. To make it available for the manufacture of soap, it has to be carefully purified with hydrochloric or sulphuric acid, after which it has a more or less yellow-brownish color. A disagreeable odor, however, adheres even to the purified fat, which does not disappear by saponification.

To obtain the fat contained in the fibrous residue, called "skutch," remaining in the glue pans, J. A. L. Leblanc, of Lyons, recommends putting the skutch in a boiler with water sufficiently acidulated with sulphuric acid to show 5° to 6° B. (13 gallons to 220 pounds of skutch). The whole is then boiled by introducing steam into a coil pipe in the kettle, and sulphuric acid of 48° B. is gradually added until complete saturation is effected or the fat floats upon the surface and no more effervescence takes place. This treatment lasts about two or three hours. The mass, in quantities of 35 to 45 pounds, is then enveloped in cloths and placed in a hydraulic press, each package being separated from the other by a plate. The expressed liquid is brought into a reservoir and the fat floating on the surface collected. The press cakes remaining in the cloths consist of substances very rich in nitrogen, and possess the full fertilizing value of the waste treated.

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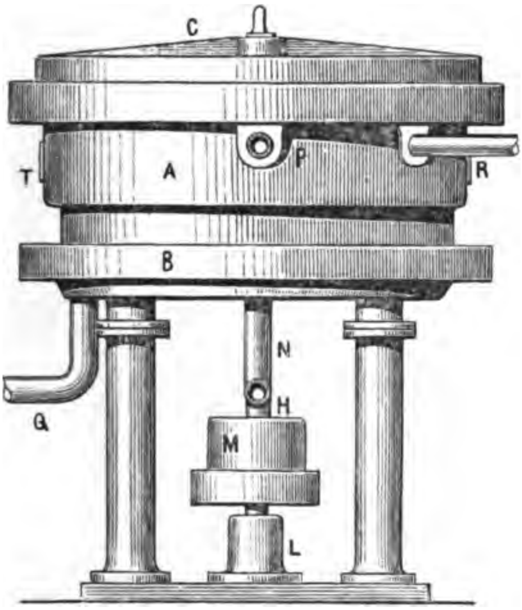
As the waste fats regained from used fats are closely related to wool fat, bone fat, and glue fat, we will here describe a few machines used in obtaining them.

The object of Thomas J. Mulling's process is the extraction of oil and fat, as well as oily and fatty substances from wool, woolen yarn, woolen tissues or similar fabrics, polishing rags, etc., or from fabrics injured in color or quality. This solvent retained in the substances treated has to be finally regained by vaporization at a higher temperature.

FIG. 251.



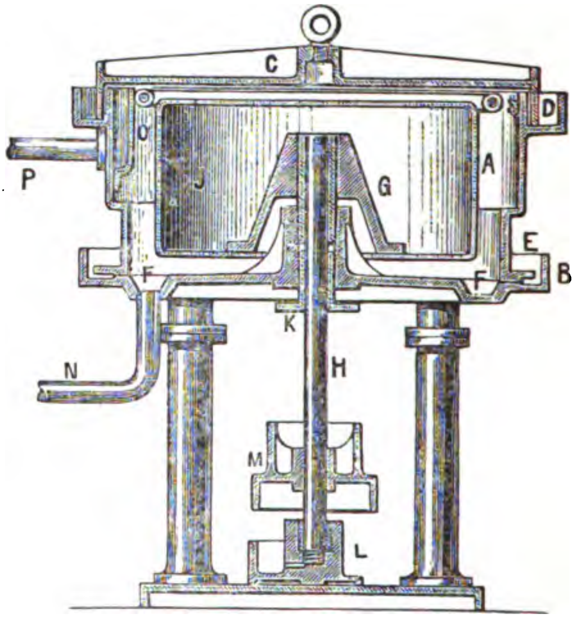
MULLING'S FAT-EXTRACTING APPARATUS.

The operation is therefore effected without the use of heat, a centrifugal drying apparatus (Figs. 251, 252 and 253), in which the use of a lubricant for the moving shaft bearings is entirely avoided, being used.

The apparatus is as follows: A cylindrical vessel *A* is secured by its bottom plate *B* upon four columns. The bottom plate *B* and the lid *C* are ground on, fastened, and hermetically closed by a water-joint, which is formed by the gutters *D* and *E*, filled with water. To keep the vessel itself dry, the collecting gutter *F*, on the bottom plate *B*, receives any water which may penetrate through the joint between *A* and *B*.

The bottom plate *B* carries a cone *G*, which forms the bearing for the vertical shaft *H*. Upon the latter sits a perforated basket *J*, open on top and so arranged that it can be lifted and lowered and refastened upon the shaft *H*. The latter projects from the vessel through the stuffing box *K* and runs in the step-bearing *L*, with water as a lubricant. The shaft is set in motion by the pulley *M*. The basket *J* is filled with the substance to be extracted, and fastened to the shaft *H*. The lid *C* is then closed and the solvent (carbon disulphide) ad-

FIG. 252.

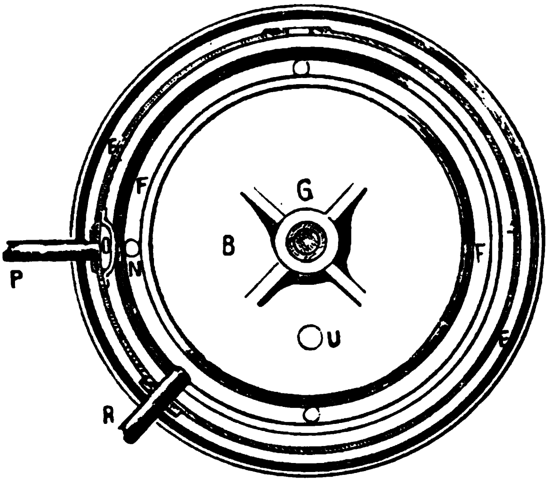


MULLING'S FAT-EXTRACTING APPARATUS. SECTION.

mitted through the pipe *N*. The solvent ascends through the substance to be cleansed, and after extracting the oily and fatty substances runs over the edge of the collecting vessel *O* and through the pipe *P* into a reservoir or a distilling apparatus for the separation of the fat.

The operation is continued until the substance under treatment is entirely freed from oily and fatty constituents, which is recognized by a sample, taken from the cock on the pipe *P*, leaving no fat stain upon blotting-paper.

FIG. 253.



MULLING'S FAT-EXTRACTING APPARATUS. GROUND-PLAN.

*A*, cylindrical vessel; *B*, bottom plate; *C*, lid; *D E*, gutters for water; *F*, collecting gutter; *G*, cone with brasses; *H*, vertical shaft; *J*, basket; *K*, stuffing box; *L*, step-bearing; *M*, pulley; *N*, pipe for solvent; *O*, collecting reservoir; *P*, outlet pipe for the solvent; *Q*, discharge-pipe; *R*, pipe for water; *S*, perforated pipe; *T*, air-pipe; *U*, discharge-pipe for air.

The pipe *N* is then closed and the pipe *Q*, which, like *N*, enters the collecting channel *F*, opened. The solvent remaining in the vessel runs through *O* into a reservoir. To assist the process the shaft *H*, and thereby the basket *J*, is set in rapid motion, which forces the solvent from the substance.

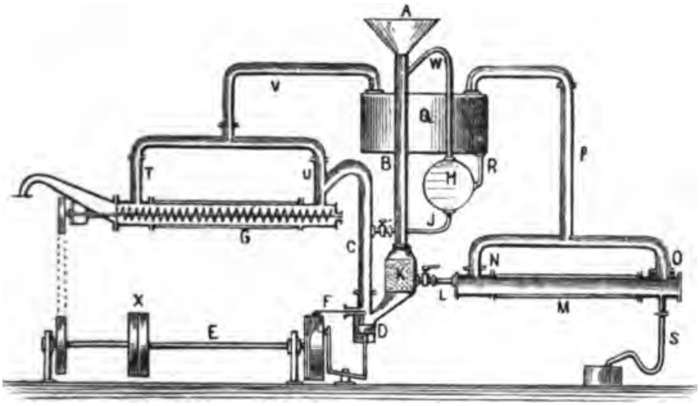
After this operation water is introduced under pressure through the pipe *R*, which leads in the vessel *A* to a perforated pipe *S*, the basket remaining at a stand-still. The perforated pipe *S* now throws a continuous fine spray of water upon the substance, washing it thoroughly, the resulting fluid running off through the pipe *Q*. The basket is then again revolved until the substance is dry. Drying can be accelerated by introducing a current of air through the pipe *T*. The air escapes afterwards through *U*. All pipes are provided with valves or cocks and constructed of lead or galvanized iron to prevent oxidation. The basket and the interior of the vessel are coated with lac varnish.

*Fricke's continuously-working fat-extracting apparatus*, Fig. 254 is well adapted for the treatment of residues from the distillation of fats which still contain 15 to 20 per cent. of oil, saw-dust used for filtering in oil refineries and retaining, after pressing and boiling (which is very inconvenient), 20 to 25 per cent. of oil, residues from oil refineries, and old axle grease. The residues from oil refineries, which contain 40 to 50 per cent. of oil and fatty acids, must first be treated with boiling water to decompose the sulphuric acid, and the axle grease be carefully treated with sulphuric acid. Like the residue from distillations of fat, they are then mixed with saw-dust and extracted with carbon disulphide, which best dissolves oil already converted into resin.

The mass to be treated is poured into the funnel *A*, and, after filling the vertical pipe *B*, slides through an inclined channel into the vertical pipe *C* over the piston *D*. The latter, put in motion by the shaft *E* by means of a crank-pin and lever, lifts up the mass, which is caught by the slide *F*, moved backward and forward by the shaft *E*. As the piston furnishes constantly fresh material which is caught by the slide, the pipe *C* is entirely filled to the top. Here the mass falls into the horizontal pipe *G*, and is conveyed by a transport screw moving in it to the obliquely ascending mouth of the pipe where it leaves the apparatus.

While thus traversing the apparatus, the mass is freed from fat by carbon disulphide, which enters the pipe *C* through the pipe *J* from the reservoir *H*, and after penetrating and freeing the mass from fat, ascends the oblique channel and passes through the filter *K* and the pipe *L* into the jacketed pipe *M*, which is heated by steam. Here the carbon disulphide is vaporized, the gas passing through the bends *N* and *O* and the joint-pipe *P* into the condenser *Q*. It leaves the latter in a liquid state and passes through the connecting pipe *R* into the reservoir *H* to re-commence its circuit.

FIG. 254.



FRICKE'S FAT-EXTRACTING APPARATUS.

*A*, funnel; *B*, filling pipe; *C*, vertical pipe; *D*, piston; *E*, driving shaft; *F*, slide; *G*, horizontal pipe with transport screw; *H*, reservoir; *J*, connecting pipe between *H* and *C*; *K*, filter; *L*, connecting pipe; *M*, evaporating pipe; *N O*, bends of *M*; *P*, discharge-pipe; *Q*, condenser; *R*, connecting pipe between *H* and *Q*; *S*, discharge pipe for fat; *T U*, bends of *G*; *V*, discharge-pipe; *W*, air-pipe; *X*, pulley.

The fat obtained after distilling off the carbon disulphide is discharged through the pipe *S*, which is bent like a siphon to exclude the atmospheric air and facilitate the discharge of the fat, which would be difficult on account of a small vacuum formed by the condensation of the carbon disulphide.

The carbon disulphide retained in the mass freed from fat is regained by heating the jacketed pipe *G* with steam, so that the carbon disulphide is vaporized and, after passing through the bends *T* and *U* and the joint-pipe *V*, runs through the pipe *R* into the reservoir *H*. From the latter, the pipe *W* leads into the pipe *B*, so as to utilize any ascending gases of carbon disulphide for absorption into the mass filling the pipe *B*.

The shaft *E* is driven by the pulley *X*.

*Dègras, Sod oils* (*Gerberfett, Lederfett*, G.; *dègras de peaux*, F.) In tawing, which is the oldest method of preparing leather, the skins are treated with fat or oil. After the necessary preparatory operations, the oiled skins are piled up in a conical heap in a heated chamber, where they undergo a sort of fermentation.

Notwithstanding this fermentation, which effects the splitting of the fats into fatty acids and glycerin, a portion of the oil—about 50 per cent. of that originally used—is contained in the skin in an unaltered condition and can be regained mechanically by wringing and pressing. The fat thus obtained forms the best quality of dègras. The remaining fat has to be removed by warm potash solution, whereby it is partly saponified. The white liquor running off separates, on standing, a fatty mass, which forms the second quality of dègras, and is used for greasing upper leather, etc. Or the fat is regained by compounding the above oily mass with sulphuric acid.

While formerly dègras was obtained only as a by-product in tanning and currying skins, a large quantity of an artificial product is at present manufactured in France and Germany, especially in Paris, Cologne and Worms. The materials required for the purpose are: Natural dègras, train-oil, wool fat, palm oil, tallow and caustic marble.

In commerce pure dègras is also known as ***mollo*** or ***möellon***. The fats obtained in tanneries, however, do not supply the demand, and for the manufacture of artificial dègras möellon has, therefore, to be produced by artificial means. This is done by repeatedly saturating skins of less value with train oil and washing them with potash solution until rent to rags. The dègras thus produced, of course, does not differ in any respect from that obtained in tawing, the conditions for the alteration of the fat being the same.

In French factories the operation is generally carried on as follows: Train oil together with about 2 per cent. of its weight of caustic marble is brought into a boiler and heated to boiling, this temperature being maintained, with constant stirring, for about one hour. Since the mass foams very much, allowance must be made for a space for rising, and the operation of stirring has to be so conducted as to exclude scorching and consequent darkening of the mass. Caustic marble being rather expensive, it has been endeavored to use freshly burnt lime as a substitute. The latter must, however, be converted into hydrate with 50 per cent. of 10° Be. soda lye.

In heating the train oil with lime a lime-soap is formed, which is, however, held in solution by the remaining fat. The fatty mass is strained through linen to retain particles of lime, then brought into a tank and allowed to cool to 113° F. About one half the quantity of water of 86° F., as pure as possible, is then added in small portions. After reposing for 24 hours the mass, as regards consistency, resembles soft butter. A mixture consisting of one half natural dègras and one-half crude wool fat, train oil, common salt and palm oil is then mixed, at 104° F., with the same quantity of the lime soap, and the mixture stirred until cold. With correct proportions of the separate fats an excellent product is obtained, but the best mixtures are kept secret by some factories.

A Frankfort firm uses a dilute potash solution instead of lime. The filling materials consist mostly of magnesium chloride solutions, which have the advantage of the dègras penetrating readily into the leather and keeping it moist.

The patented process of Schill & Seilacher, of Stuttgart, consists in atomizing the train oil heated to about 248° F. in a chamber by means of hot air. A portion of the atomized oil precipitates again in the chamber, whereby an odorless fat is obtained. However, the greater portion of the fat in the form of mist escapes from the chamber, and is condensed in adjoining chambers which are provided with cross-partitions offering much resistance. The product here obtained is more oxidized than that obtained in the first chamber, and is mixed with it, and thus brought into commerce.

Commercial dègras contains considerable quantities of water (generally 12 to 20 per cent.) which, with a good product, must not separate even after standing for some time. The emulsive power of dègras is dependent on the quantity of a resin-like substance formed by oxidation. The more of this substance is present, the more readily emulsion takes place, so that a dègras with 13.9 per cent. of it gives with 53 per cent. water such an intimate mixture that it remains perfectly homogeneous after two months.

This resin-like substance is brown, and fuses at 149° to 152.6°F. It is saponifiable, but cannot be precipitated from an alkaline solution by common salt, differing in this respect from the fats. It is insoluble in water, soluble in alcohol and ether, but insoluble in petroleum-ether, differing in this respect from resin. It does not occur in train oils, but is first formed in tawing.

Simand calls this resin-like substance dègras-maker ("degrasbildner"). In a crude state it is black-brown, and pale brown when purified. It is readily soluble in caustic alkalies and ammonia, and can be precipitated from these solutions by acids. It is perceptibly soluble in hot, especially in acidulated, water; it is soluble in alcohol, glacial acetic acid, and aniline; almost insoluble in ether, especially in petroleum-either and benzole; it does not fuse without decomposition, and occurs chiefly in genuine dègras and in larger or smaller quantities in nearly all kinds of train oil, old oils containing more than fresh, and dark more than pale.

In dègras the dègras-maker is not present in a free state, but contained in a saponifiable combination, which, contrary to the dègras-maker, is, in a free state, soluble in petroleum ether, but soluble with difficulty in alcohol.

Simand considers the dègras-maker a nitrogenous body, which is, however, disputed by Fabrion, who considers dègras a mixture of oxyfatty acids.

Most kinds of dègras contain non-saponifiable substances derived from the train oils. Thus, for instance, dègras from sperm whale oil contains much cetylic alcohol, dègras from liver oil an oily non-saponifiable mass, of which unchanged liver oil contains 6 per cent.

The content of free fatty acids varies very much, its magnitude being, however, without influence upon the quality of the product.

Genuine dègras is contaminated with solid organic sabstances —particles of skin and membranes—which, however, should not exceed 5 per cent. The specific gravity—0.945 to 0.955—of dègras freed from water is greater than that of the train oil—0.927 to 0.930—used in its preparation.

An examination of train oils, according to Livache's process, has shown that the train oils which absorb most oxygen are most suitable for the production of dègras. Whale oil deserves the preference, while sardine oil, which yields only 3 to 4 per cent of resin-like substance, is unsuitable for the purpose.

*Analysis of dègras. Determination of water.* 5 grammes of dègras are mixed with sufficient calcined quartz sand to form a solid nearly dry mass, and the latter is dried at 248° F., and weighed.

*Fat and insoluble substances.* Dilute 20 grammes of the sample with petroleum-ether, and filter through a previously dried and weighed tube closed by a cotton stopper. Distil off the filtrate, bring the residue into a dish, evaporate, dry at 248° F., and weigh. For the determination of the undissolved portion, dry the filter-tube at 248° F., and weigh. Next incinerate the contents of the tube in a platinum crucible. If only an inconsiderable residue remains behind, organic substance alone was present; but if the residue is large, it is weighed and analyzed (clay, chalk, sulphate of lime, etc.).

*Ash.* For the determination of ash 5 grammes are used.

If the ash is alkaline, extract it with water, filter and titrate. Simand weighs out 25 grammes of dègras in a platinum dish, places the latter upon an asbestos plate and heats, stirring constantly, until the water is expelled. The rod is cleansed with a piece of filtering paper, the latter thrown in the train oil and used as a wick for burning the fat. The residue is finally incinerated. Varieties of dègras containing ferrous oxide make the leather gray and hence the ash is chiefly tested for it. Simand found the effect of as small a quantity as 0.05 per cent. ferrous oxide very remarkable. However, such dègras can be improved by adding to 220 pounds of it 1 pint of a 1 per cent. oxalic acid solution.

*Mineral acids.* If the dègras shows a strong acid reaction, boil 25 cubic centimeters of the sample with 200 c.c. of water and allow to cool; then separate the two layers by means of a separatory funnel, determine in one portion of the watery layer the nature of the acid (mostly sulphuric acid), and titrate another aliquot portion (50 cubic centimeters) with soda lye.

*Fibres of skin and leather.* Shake the dègras repeatedly with petroleum ether, wash the residue, which contains only water, sand, soap and the leather fibres, first with water then with alcohol; dry, weigh, incinerate and deduct the weight of the ash from the total weight.

*Resin-like substance.* The soap solution extracted with ether for the determination of the non-saponifiable portion is evaporated to expel the ether remaining in solution, and precipitated while hot with pure common salt in excess. When cool, filter the colored fluid from the separated soap into a flask and mix with hydrochloric acid, whereby the resin-like substance separates in flakes, which are combined by boiling and settle on the sides of the flask. Allow to cool, and by shaking with ether, evaporating and drying, bring the mass into a state to be weighed.

For the determination of the resin-like substance—the dègras-maker—Simand proceeds as follows: Saponify, according to the content of water, 20 to 25 grammes of dègras in an

Erlenmeyer flask with funnel, with 5 to 6 grammes of solid caustic soda, previously dissolved in 10 cubic centimeters of water, and 50 to 60 cubic centimeters of alcohol Remove the funnel, drive off the alcohol, dissolve the soap in distilled water, and separate the fatty acids with hydrochloric acid. Heat until the fatty acids float clear upon the surface and the dègras-maker collects in lumps; allow to cool and separate the acidiferous water from the fatty acids. Since the water may contain some dègras-maker in solution, neutralize with ammonia and evaporate. Wash several times by boiling with water, and combine the wash-waters neutralized with ammonia with the first sub-lye. Dissolve the residue from evaporation in a little water, slightly acidulate the solution with hydrochloric acid, filter off the small quantity of dègras-maker precipitated, wash, dry and return it to the Erlenmeyer flask, the contents of which have in the meanwhile been dried at 221° F. Next extract repeatedly with 100 to 120 cubic centimeters of petroleum-ether, which dissolves the fatty acids, the dègras-maker and a small quantity of albumen remaining behind. The residue is dissolved in alcohol with the assistance of heat and filtered off from the albuminous substances. Distil off the filtrate and weigh the residue as dègras-maker. The petroleum-ether extracts may be distilled off and used for the determination of the quantity of fatty acids, their fusing point, etc.

A dègras is pure and genuine only when it contains at least 12 per cent. dègras-maker referred to content of water of 20 per cent. in the dègras. The content may also rise to 16 or 17 per cent. The method is accurate to 0.5 per cent.

*Free fatty acids.* Titrate with soda lye and phenolphthalein. For whale oil the calculation of the percentage of free fatty acids is based upon the saponification number 159. The oil from dègras contains mostly 15 to 19 per cent. free fatty acids.

Jean gives the following examples of the composition of dègras:

| | 1. | 2. | 3. | 4. | 5. | 6. | 7. |
|---|---|---|---|---|---|---|---|
| Water .......... | 18.90 | 14.84 | 12.93 | 28.90 | 19.20 | 5.39 | 8.90 |
| Ash ............ | 0.25 | 0.13 | 0.55 | 0.70 | 0.07 | 0.25 | 1.21 |
| Fragments of skin. | 0.30 | 0.30 | 0.09 | 0.58 | 0.27 | — | 1.59 |
| Oils ............ | 69.71 | 74.65 | 80.00 | 66.93 | 75.06 | 84.87 | 72.15 |
| Non - saponifiable matter......... | 6.84 | 6.05 | — | — | — | — | — |
| Resin - like substance ........ | 4.00 | 4.05 | 5.81 | 3.52 | 4.80 | 9.46 | 16.15 |

Simand obtained the following results:

| | | Quantity of dègras-maker. Per cent. | Fusing point of the separated fatty acids. | Soap. Per cent. | Leather fibres referred to dègras containing water. Per cent. | Content of water. Per cent. |
|---|---|---|---|---|---|---|
| Dègras according to the French method, free from water. | 1 | 19.14 | 74.4°-83°F. | 0.73 | 0.07 | 16.5 |
| | 2 | 18.43 | 83°-84.2°F. | 0.49 | 0.12 | 20.5 |
| | 3 | 18.10 | 87.8°-89°F. | 0.68 | 0.18 | 12.0 |
| Natural dègras, free from water. | 4 | 20.57 | 92°-93.2°F. | 3.95 | 5.7 | 35.0 |
| | 5 | 18.63 | 81.5°-82.4°F. | 3.45 | 5.9 | 28.0 |
| | 6 | 17.84 | 82.4°-83°F. | 3.00 | 4.5 | 30.5 |

*Artificial dègras.* The presence of foreign fats, such as wool-fat, olein, tallow, may, according to Jean, be presumed if the specific gravity of the oil extracted from the dègras is less than 0.920, while dègras from fish and whale oils shows a specific gravity of 0.949 to 0.955. For the presence of tallow the fusing point of the fatty acids is characteristic. The fatty acids of tallow fuse only at above 104° F., and hence raise the fusing point of the fatty acids of dègras, the acids from whale oil fusing at 76.8° F., those from liver oil at 65.3° F., and those from Japanese sardel oil at 96.4° F. Admixed resin is found by extracting with petroleum ether, which does not dissolve the resin-like substance contained in dègras.

In the examination of artificial dègras Simand takes into consideration, besides ash and water: 1. The dègras-maker, which possibly may be derived from an addition of small quantities of genuine dègras or from the train oils; 2. Wool-fat; 3. Vaseline oil; 4. Resin.

For the determination of the dègras-maker Simand proceeds in the same manner as with genuine dègras, excepting that he extracts the fatty acids with ether instead of petroleum ether, because the fatty acids of wool-fat remain dissolved in ether in the cold.

For determining wool-fat drive off the ether solution from 5 to 6 grammes of dègras in a weighed flask, boil the residue with one and a half times the quantity of acetic acid, dry the washed fat, and dissolve it in fifteen times the quantity of boiling alcohol. By cooling, cholesterol ether in crystals is separated. The latter is twice recrystallized from 15 times the quantity of alcohol, dissolved in ether, the ether distilled off, and the residue weighed. Wool-fat yields on an average 14.05 per cent. of cholesterol ether. By multiplying the weight of the crude cholesterol ether with 14.5, a rough approximation of the content of wool-fat is obtained.

A content of wool-fat may also be recognized by the lustrous surface of the solidified fat, or if it does not solidify, by the lustrous non-crystallizing surface of the fatty acids separated after crystallization. The characteristic odor of wool-fat also appears by rubbing a small sample upon the hand.

*Vaseline oil and rosin.* The alcoholic filtrates of cholesterol ether are distilled off to a small residue, the latter saponified with caustic soda, the solution mixed with glycerin or alcohol, and extracted with petroleum ether, whereby the vaseline oil passes into solution. The soap solution is decomposed with acid, and the separated fatty acids are dissolved in alcohol. The solution is brought into a weighed flask, the alcohol driven off, the residue weighed, and the content of rosin determined in an aliquot portion.

*Fuller's fat* (*Walkfett*, G.; *graisse de foule*, F.). The use of soaps, as well as fatty oils, in fulling cloths and woolen goods in the silk and cotton industries, dye-houses, etc., has attained such enormous dimensions that the regaining of the fats contained in the waste soap-waters is a matter of considerable interest, independent of sanitary considerations.

It is calculated that in Europe alone 500,000 tons (1 ton= 2200 pounds) are annually used in fulling, which would give about 100,000 tons of lime soap, worth about $5,000,000.

In many places the regaining of fat from soap waters has been advantageously carried on for more than sixty years. The method consisted, and even yet consists, almost exclusively in decomposing the soap water with a strong acid such as hydrochloric or sulphuric acid.

By this method the separation of fat, assisted by heat, takes place largely in the form of a cream-like mass, the purification of which causes considerable loss of fat. And, besides, the already brownish fats acquire a still darker color by this purification, which has to be done over an open fire, and the aqueous vapor saturated with volatile fatty acids arising during the operation is a source of great annoyance to the neighborhood, and not without injurious influence upon the health of many persons.

To overcome these evils Vohl has proposed the separation of the fats by means of calcium chloride, and with an addition of quicklime to convert the lime salts formed by distillation into a new volatile illuminating material which he has named "Oleon."

In consequence of the general use of petroleum for illuminating, the manufacture of oleon is no longer of any value, but the precipitation of the soap-waters with a lime salt for regaining the fatty acids is of importance.

The soap-waters are compounded with an aqueous solution of calcium chloride until no more cheese-like precipitate is formed. The formed lime soap is then separated by filtration through large baskets lined with coarse hemp-cloth, and then freed from the larger portion of water by draining and pressing.

The drained mass is then brought into large vats provided with covers, and decomposed with a corresponding quantity of hydrochloric acid as free as possible from sulphuric acid. Decomposition is accelerated by direct introduction of steam, and the separated fatty acids obtained in a liquid state. The gases and vapors evolved during decomposition by the introduction of aqueous vapors pass through a cooling pipe into an iron box hermetically closed. The box contains slaked lime, and is connected by pipes with the fire-box of the steam boiler. By this arrangement all noxious gases and vapors are completely removed and destroyed.

After the decomposition of the lime soap the mixture is allowed to rest six hours. The calcium chloride solution is then drawn off through a cock in the bottom of the vat, and can be again used for another precipitation. The fatty mass is then once more mixed with half the quantity of dilute hydrochloric acid used for the decomposition, and steam introduced for ½ to ¾ of an hour.

After the fatty mass has separated from the acid aqueous fluid, the clear dilute acid is drawn off, without, however, allowing any portion of the emulsive layer to run off. This emulsive layer offers the greatest difficulty in separating the fatty acids from the aqueous fluid.

The purification of the fatty acids is either effected by simply freeing them from water, or by bleaching after dephlegmation and separation of the solid fatty acids from the liquid. If the fatty acids are not to be immediately used in the manufacture of soap, but to be brought into commerce as fats, dephlegmation is necessary. For this purpose the emulsive oil layer is either heated with an addition of common salt (for inferior quality of fat) over an open ore, or, for better qualities, by steam circulating in iron or copper pipes on the bottom of the boiler. The latter method of heating is especially used for soap waters derived from boiling silk or Turkey-red dyeing, the greater portion of which is composed of olive-oil soap.

It is frequently of advantage to bleach the fatty acids obtained, as they acquire thereby a greater commercial value, and lose, to a large extent, their disagreeable odor. The methods required will be found in *the chapter on bleaching oils, etc.*

In many cases it is desirable to separate the solid from the fluid fatty acids. For this purpose the warm bleached fatty acids are brought into large vats about nine feet high and three feet in diameter, and gradually cooled to 48° F.

To effect as complete a separation as possible very slow cooling is absolutely necessary, as otherwise the solid fatty acids do not form conglomerates of large crystals, but remain suspended in the fluid as a coagulum very difficult to separate. If the operation is conducted in a correct manner, the solid fatty acids separate on the sides and bottom of the vat in crystals resembling cauliflower. The fluid portion is separated from the solid mass through a cock in the bottom of the vat. The solid masses can be at once brought into cold presses, and are used in the manufacture of candles.

The substances obtained from the above-mentioned waste are not neutral glycerin combinations, but actual acids, and have a strongly oxidizing effect upon metals and their alloys —copper, zinc, etc.

Fuller's fat is brought into commerce in barrels, and varies very much in consistency and color. It is sometimes soft like grease, and, again, so viscous as to be difficult to remove from the barrel. The first variety melts at 86° to 95° F., and the other at 122° F. Its color, which, however, varies very much, is generally a dirty yellow brown.

For a lubricant fuller's fat is entirely unfit, as it consists principally of free fatty acids, and it is, therefore, exclusively used, mixed with other fats, in the manufacture of soft soap and rosin soap.

# CHAPTER XVII.

## BLEACHING OF FIXED OILS AND FATS.

THE object of bleaching oils and fats is, on the one hand, to give them a greater commercial value, and, on the other, to remove coloring substances which would prove annoying in their use, as, for instance, in the manufacture of light oil varnishes from linseed oil.

The oldest and simplest method of freeing oil from coloring matter is based upon the action of light and air, which might be called the "natural or sun process" as distinguished from the "chemical or quick process," which was first introduced, in 1786, by Berthollet. He used chlorine, discovered by Scheele, which since then has, however, been supplemented by other chemical agents.

Generally speaking, light acts chemically by re-separating oxygen from various substances, or by promoting the combination of the oxygen of the air with the hydrogen and carbon of the organic substance, *i. e.*, the coloring substances, whereby the latter are frequently entirely decomposed or changed to a lighter hue. In many cases the actual effect of light may be due to the fact that under certain conditions it promotes the formation of ozone or of peroxide of hydrogen, which oxidizes coloring substances with greater ease than the ordinary oxygen of the air. Sunlight is, of course, the most powerful bleaching agent.

For chemical bleaching various means are used, but all depend upon the action of four agents—ozone, peroxide of hydrogen, chlorine, and sulphurous acid.

Peroxide of hydrogen $\left.\begin{matrix}HO\\H\end{matrix}\right\}O$ and ozone $\left.\begin{matrix}O\\O\end{matrix}\right\}O$ exert an oxidizing effect upon the coloring substances; chlorine withdraws from them, on the one hand, hydrogen under the formation of acid, while, on the other, chlorine is substituted for hydrogen; sulphurous acid unites with the respective coloring substance to a colorless combination.

For bleaching by means of light, especially sunlight, the respective oils are exposed to the rays of the sun in large cylindrical glass vessels or zinc boxes or wooden boxes lined with tin plate. The layer of oil should not be deeper than one-and-a-half to two inches.

To accelerate bleaching by sunlight, chemicals are frequently added. It is, for instance, of great advantage to add oil in bottles, such as olive oil, colza oil, linseed oil, a small quantity of 90 per cent. alcohol. By frequent shaking the effect is perceptible in a few weeks. Or the oils are compounded with solution of copperas before exposure to the sun, and frequently shaken.

Thus, for instance, for bleaching linseed oil, the oil in portions of about 18 pounds each is distributed in glass balloons, holding about 4½ gallons each. Next prepare a solution of 220 pounds of copperas in about 50 gallons of rain-water and add 5 to 6 quarts of it to every balloon. Expose the balloons to the direct light of the sun and shake frequently, at least once a day. The time required for bleaching varies according to the temperature and the intensity of the sunlight.

Besides, by means of sunlight, oils are very quickly bleached in rooms holding air containing ozone, which is produced by means of electricity or by placing moist phosphorus sticks in the room.

Peroxide of hydrogen, which is now manufactured on a large scale and brought into commerce in a 10 per cent. solution, is well adapted for bleaching oils, since it only requires frequent agitation of the solution with the oil to be bleached. Bleaching is effected in a few days, the oils clarifying rapidly, so that their separation from water by means of a siphon is readily accomplished. According to the degree of coloration, 4 to 5 per cent. of the 10 per cent. solution of peroxide of hydrogen suffices for all purposes, except, perhaps, for linseed oil and camelina oil, where somewhat more has to be used.

The bleaching of oils by treating them with potassium permanganate and sulphuric acid is based upon the action of ozone. The process is conducted in wooden vats lined with lead and provided with a stirring apparatus and heating pipe. Solution of chromic acid or potassium permanganate strongly acidulated with sulphuric acid is gradually added to the oil, with constant stirring, and the stirring continued one-half to one hour, though frequently the process is finished sooner. After resting six to twelve hours the oil has clarified over the greenish or brownish fluid containing chrome alum or manganese alum. The acid fluid is then drawn off, the oil washed twice or three times with warm water and allowed to rest. The clarified oil is then removed, an emulsive layer remaining between the aqueous layer and the clear oil, especially with fat. By compounding this emulsive layer with 10 to 15 per cent. of petroleum-ether immediate separation is effected. The petroleum-ether is regained by distillation when, after several bleachings, sufficient material to fill a still has been obtained, and again employed for further operations.

For 220 pounds of oil or fat about ½ to ⅔ pound of potassium bichromate or potassium permanganate is used, and double the quantity of sulphuric acid, previously diluted with 5 to 6 times the quantity of water.

The same object is obtained by mixing 220 pounds of oil or fat with about 2 pints of sulphuric acid previously diluted with about 7 to 8 gallons of water and gradually adding to the heated mixture small portions of very finely pulverized pyrolusite until the mass, at first black, becomes white. After completion of the operation the oil or fat is washed, and further treated as above.

To bleach with chlorine, chlorine is either developed as much or as combination in the form of potassium or sodium hypochlorites; the first known as "Eau de Labarraque" and the latter as "Eau de Javelle," are used. These solutions should contain no free sodium carbonate originating from their preparation, as otherwise an emulsive mixture difficult to separate is formed. Oils, such as linseed, colza oil, etc., frequently shaken with these solutions, are thoroughly bleached, but they retain a peculiar chlorine-like odor, which, however, can be almost entirely removed by adding to the second wash-water a small portion of hydrochloric acid; the latter must, of course, be removed by a third washing.

For bleaching large quantities of oils or fats with chlorine, wooden vats, provided with a wooden stirring apparatus and a heating pipe coated with rubber or tar, are used. The chlorine is developed from hydrochloric acid by the addition of substances rich in oxygen, such as potassium permanganate, sodium hypochlorite, and calcium hypochlorite. Two hundred and twenty parts by weight of oil are first mixed with 2½ to 5 parts of crude hydrochloric acid previously diluted with four times the quantity of water; ¾ to 1 part of the above-mentioned salts rich in oxygen is then gradually added. Or the process may also be carried on in a reverse order by first mixing the solution of the salt with the oil and then gradually adding the hydrochloric acid in small portions. In using "Eau de Javelle" or solution of calcium chloride, 1 part by weight of it is allowed for 1 part of hydrochloric acid.

Palm oil and palm-nut oil were first bleached with potassium bichromate and hydrochloric acid, in 1859, by Englehardt, of Leipzig.

Pyrolusite can also be utilized to the same advantage as the above combinations.

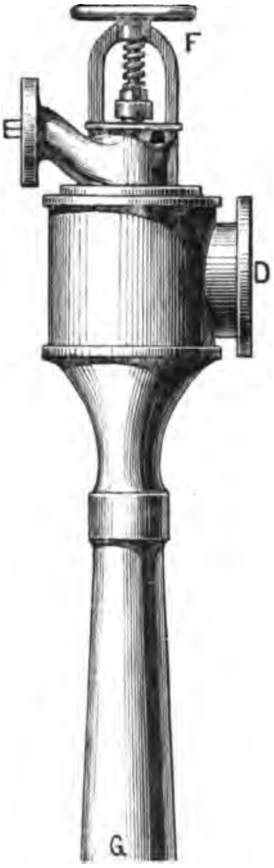
In all manipulations care must be had that the hydrochloric acid predominates. For washing observe the rules given above.

Instead of hydrochloric acid, a mixture of sulphuric acid and sodium chloride, from which hydrochloric acid is developed, may be used.

The principal objection to the use of chlorine is that it attacks the oils and fats very much by readily decomposing stearic acid, and every excess of chlorine, which can never be entirely avoided, must therefore injure the quality of the fat.

For bleaching with sulphurous acid, $SO_2$, the cheap commercial bisulphite of sodium, $NaHSO_3$ is used. A good result is already produced by agitating a concentrated solution of it with the oil, but to obtain the effect of all the acid in the solution, it is compounded with dilute sulphuric acid. The vats lined with lead, etc., already described, are used. 1 to 1½ parts by weight of bisulphite of sodium suffice for 220 parts of oil. Here also care must be had to add the sulphuric acid gradually until it is slightly in excess, as by adding too much of it at a time the development of sulphurous acid takes place too vigorously, and escapes without effect. Koerting's air-suction or steam-jet suction apparatus is well adapted for bleaching with sulphurous acid.

FIG. 255.

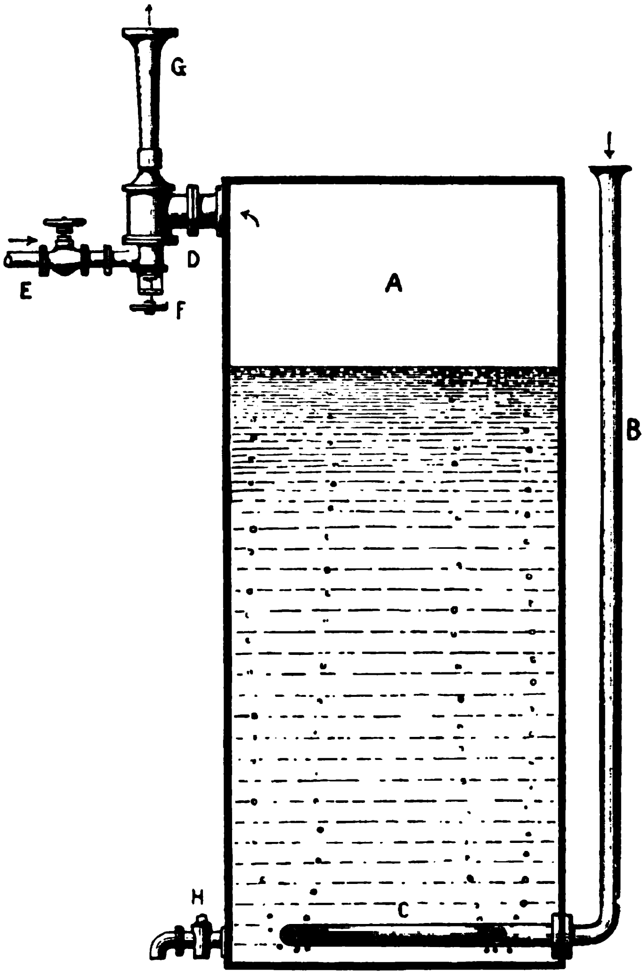


STEAM-JET SUCTION APPARATUS.

The steam-jet suction apparatus (Fig. 255), manufactured for this purpose of lead, acts by steam entering the apparatus through the pipe *E*, and passing in the interior through a series of conical nozzles. In passing these nozzles the exterior air is very rapidly sucked in through *D*, and expelled through the opening *G*.

The steam-jet suction apparatus can act, as wanted, either by suction or pressure, and, therefore, rarefy or compress the air as may be required. It is so constructed that, with a tension of three atmospheres of steam, it can with suction overcome the pressure of a column of water of 10 to 26 feet, and with pressure one of 10 to 13 feet.

FIG. 256.



STEAM-JET APPARATUS FOR BLEACHING WITH SULPHUROUS ACID.

*A*, oil vessel; *B*, suction-pipe for sulphurous acid; *C*, perforated pipe; *D*, connecting joints; *E*, steam pipe; *F*, regulating valve; *G*, steam passage and exit; *H*, discharge-cock.

In Fig. 256 the air-suction apparatus is placed on the top of the vessel containing the oil to be bleached. Though the pipe

*B*, and the perforated coil *C*, the apparatus sucks sulphurous acid in an extremely finely divided state through the oil until the latter is bleached; the vessel must, of course, be hermetically closed.

The sulphurous acid used for bleaching is evolved by burning sulphur under admission of air in an oven of simple construction. To regulate the process, the introduction of steam can be increased or decreased by the valve *F*.

The apparatus is very effective, and can also be used for somewhat thickly fluid fat, only it must not be too pasty.

Oil bleached by means of sulphuric acid is washed in the manner already described.

Animal charcoal is not very well adapted for bleaching oil.

Before bleaching train oil, it is best first to remove the gelatinous substances by treatment with solutions of tannin.

The following method which, it is claimed, is adapted for solid and fluid fats, has been proposed by Herzog for bleaching and clarifying oils and fats for domestic and industrial purposes. Cotton-seed oil, rape-seed oil, and all other fat oils are prepared for treatment by mixing in a large tank with 2 to 3 per cent. of common salt, and thoroughly stirring for 5 to 10 minutes, with 25 to 50 per cent. of water. After a rest of 24 to 48 hours, it will be found that a portion of the impurities and the water and salt have settled on the bottom. The supernatant oil is then drawn off into another tank, and again thoroughly washed with cold water and again drawn off after a rest of 6 to 12 hours.

This treatment with common salt is said to be especially valuable for the preparation of fine table oils, but can also be used for other oils, such as linseed oil, train oil, etc. By conducting at the same time during the mechanical treatment an electrical current through the mixture, the oil is bleached by the decomposition of the common salt by the action of the electric current and the formation of secondary combinations of strong bleaching power.

For many oils and fats which readily become rancid or spoil, it is recommended to add 2 to 3 per cent. of bicarbonate of sodium to the above process. Besides the second washing with cold water, the oil can also be treated with steam conducted through it in a finely divided state, 5 to 10 minutes being sufficient for cotton-seed oil, while 15 to 20 minutes are required for rape-seed oil, and 30 minutes for fish oil. By this treatment the constituents causing rancidity are removed, and the slimy particles precipitated.

Instead of steam repeatedly heated, air mixed with 20 to 30 per cent. of hot water may also be forced through the oil by means of a blowing engine. By filtering the oil thus prepared, and storing it for some time, a pure product of an agreeable taste and clear pale yellow color is obtained. For filtering, the ribbed sides and bottom of the filtering vessel are covered with endless filtering paper.

For the preparatory treatment of varnish oil, burning and lubricating oils etc., the oil is compounded with a solution of 2 per cent. of common salt in 15 to 20 per cent. of water of 176° to 212° F., which during the stirring is still further heated by the introduction of steam. One to 1 ½ per cent. of hydrochloric acid diluted with 15 to 20 per cent. of water is then added, with constant stirring, and finally steam is introduced at intervals of five minutes. The oil is then allowed to collect in settling tanks.

In many cases an addition of potassium permanganate or potassium bichlorate or potassium chlorate, previously dissolved in as little water as possible, is useful. For 100 parts of oil about $\frac{1}{10}$ part of the last-named salts and 1 ½ to 2 parts of common salt are used. The heated oil to be bleached is successively mixed with the salt solutions, 2 to 3 per cent. of hydrochloric acid or 1 to 1 ½ per cent. of sulphuric acid being added by means of a rose, with constant stirring, for one hour. Then add 30 per cent. of warm water to the mixture and allow it to rest.

After the oil is drawn off it is several times washed with water, with an addition of some soda, and finally treated with steam. The slimy sediment may be utilized in the manufacture of soap. This method is applicable to mineral, vegetable and animal oils.

In the laboratory of the "Ecole nationale des Arts et Metiers," at Aix, L. A. Levat has made a number of experiments to purify salad and lubricating oils by means of electricity. A cylinder was filled with an inferior quality of olive oil of an acid taste and brown color, and sufficient water added to form a layer 1 to 1½ inches deep below the oil. In this water, dipped the electrodes, which were connected with a small dynamo. By allowing a weak current of 2 or 3 volts tension to pass through until the water was nearly decomposed, an oil of pale color was obtained, which was only slightly turbid, due perhaps to water mechanically carried along. On the other hand, the taste was completely changed; the acidity had disappeared and the oil had acquired an agreeable, sweet, piquant taste. Levat subjected about 20 kinds of oil of a bad taste and dark color to the same process, and in every instance succeeded in essentially improving them. With some care the oil did not become turbid but remained perfectly bright. Levat also made a series of experiments with inferior lubricating oils containing at least 5 per cent. free acid. The content of acid decreased to 1 per cent. and by repeating the process even to $\frac{1}{10}$ per cent.; but the remainder could not be removed.

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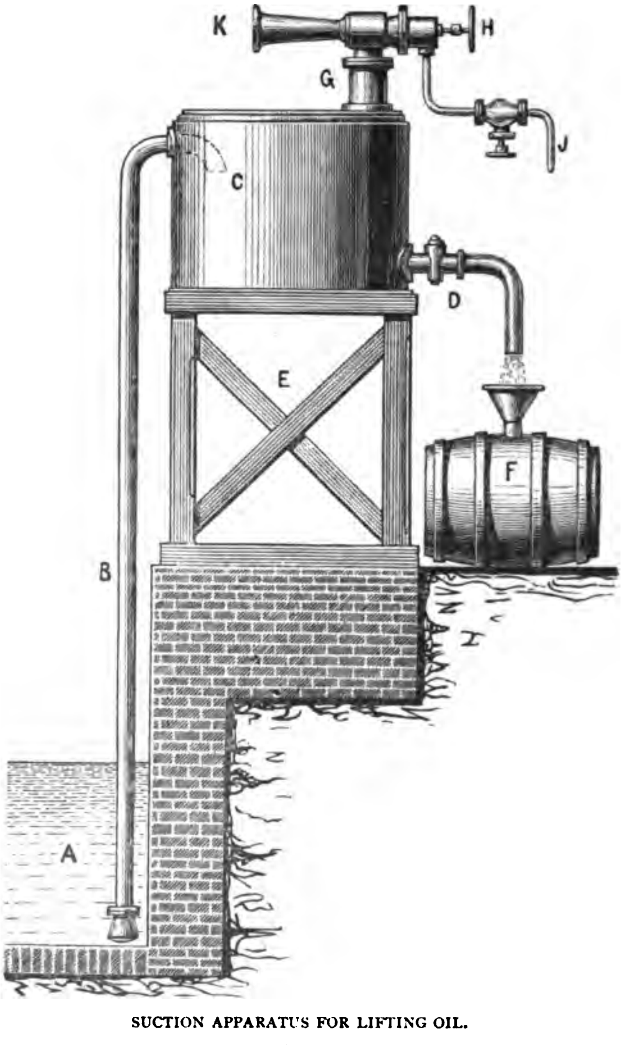
The air-suction apparatus described on p. 221 (Fig. 255) may also be used for lifting oil to a height of 26 feet. The arrangement is shown in Fig. 257.

At the beginning of the operation the steam-valve *J* is opened and the discharge-cock *D* closed, until the upper reservoir *C* is filled through the suction-pipe *B* from the oil reservoir *A*. The steam-valve *J* is then closed, and the raised oil discharged from the reservoir *C* through the cock *D* into the barrel *F*.

FIG. 257.



SUCTION APPARATUS FOR LIFTING OIL.

*A*, oil reservoir; *B*, suction-pipe; *C*, oil reservoir; *D*, discharge-cock; *E*, framework; *F*, oil barrel; *G*, connecting joint; *H*, regulating valve; *J*, steam pipe; *K*, steam passage and exit.

# CHAPTER XVIII.

## ARTIFICIAL BUTTER.

### 1. *Oleomargarine.*

AT the suggestion of the late Emperor Napoleon III., whose desire it was to furnish to the poorer classes and to sailors an article which should be cheaper and more stable in composition than ordinary butter, the French chemist Mège-Mouries, in 1869, was led to undertake the manufacture of a palatable substitute for butter from the fat of animals slaughtered for food.

Mège was employed on the Imperial farm at Vincennes. He endeavored to imitate the physiological process which he supposed takes place when the cows are insufficiently fed, and when, therefore, the butter which they furnish is derived from their own fat. From beef he obtained a "fat, which melted at almost the exact temperature of butter, possessed a sweet and agreeable taste, and which for most purposes could replace ordinary butter, not, of course, the finest kind, but which was superior to it in possessing the advantageous peculiarity of keeping for a long time without becoming rancid."

Before the breaking out of the Franco-Prussian war Mège had established a factory at Poissy. Although operations were to some extent suspended by the war, large quantities of this new article of food were used in Paris during the siege of 1870–71, and it then gained such a strong hold that at present Paris alone consumes 60,000 to 80,000 pounds daily. A patent was granted to M. Mège by the English government July 17, 1869, which was the first patent issued that has any connection with this subject. As early as 1846, William Palmer took out a patent in England for "treating fat or fatty matters from beef, mutton, veal and lamb," but the product obtained was similar to lard (and as the specifications state) "but will not have the odor, flavor, or taste of lard;" this is owing to the fact that the product is flavored with "bay leaves."

In this country it appears that a patent was granted, January 3, 1871, to W. H. Bradley, but this patent does not cover the material under the name of butter; the Bradley process made no greater claim than to make the grosser fats equal to lard for cooking purposes by a patent process. Later in the same year, November 2d, 1871, a Frenchman, W. K. Peyrous, obtained a patent under a claim to make "the finer animal fats equal to the best lard, for cooking purposes only;" this was supplemented by the further claim that the process "made these fats as easily digestible as lard and butter." Two years later, April, 1873, E. Q. Parafe obtained a patent for the manufacture of a product which under the specifications set forth in the patent was called "oleomargarine." Parafe started a large company, called the "Oleomargarine Manufacturing Company," in New York City. During the same year, December 30, 1873, Mège's process was patented in this country. Unlike the other patents this provided for the extraction of the oil from tallow, lard and other fats at a low temperature, and after mixing with milk, either sweet or sour, churning until it assumed the semblance of butter. Mège's patent, possessing as it does historical interest, is given in full:

*Hippolyte Mège, No.* 146,012, *dated December* 30, 1873.

To all whom it may concern:

Be it known that I, Hippolyte Mège, of Paris, France, have discovered a new and improved process of transforming animal fats into butter, of which the following is a full, clear, and exact description:

The butter which is obtained from milk is produced by the cow elaborating her own fat through her cellular mammary tissues at the low rate of temperature of the body.

The animal fat from which the butter-cells in milk are produced is composed chiefly of oleine, margarine, and stearine, and small quantities of other substances.

The natural process performed by the cow consists, mainly, first, in separating the oleomargarine from the stearine without developing disagreeable odors or flavors in the oleomargarine; and, secondly, in producing a slight change in the oleomargarine, by which it assumes the character of butter.

My invention, hereinafter described, is based upon a discovery made by me, that when the fat is rendered at a low temperature, considerably below that heretofore employed in the ordinary rendering of fat, it has the taste of molten butter, and does not acquire that peculiarly disagreeable flavor heretofore supposed to be necessarily attached to melted fat or tallow, and which is designated as "tallowy flavor."

I have succeeded in obtaining excellent results by rendering the crude fat at a temperature of 103° Fahrenheit, which is below the temperature at which the tallowy flavor is created. The temperature may be raised above this point in order to facilitate the operation, provided care be taken to avoid attaining the temperature at which the tallowy flavor is created.

The precise limit to which it is safe to increase the rendering temperature can be ascertained by trial under various circumstances with the different kinds of fat. The temperature must, however, be far below that heretofore ordinarily used in rendering fats when no such object as I propose—to wit, the making of a butter-like product—was had in view. I do not think it would be safe to vary many degrees above that specifically indicated.

I have also discovered that, in order to neutralize any fermentation of the fat before or during its treatment, the raw fat should, as soon as possible after the death of the animal, be plunged in a solution of fifteen (15) per cent. of common salt and one per cent. of sulphate of soda, the effect of which would be to prevent such fermentation.

In carrying out my process I first crush, grind, or disintegrate the fat by any suitable machinery, such as rollers or millstones, in order to break up the cellular tissues in which the fat is contained in the animal, and thus cause it to be more easily melted or rendered by the application of low temperatures. This fat thus disintegrated is to be slowly raised to a temperature of 103° Fahrenheit in a vessel in which the temperature can be raised at will until the rendering shall be complete. The temperature, as before stated, must be so regulated that the rendered fat will have the taste of molten butter, and care should be taken not to heat it so as to induce the change which produces the usual disagreeable taste of melted fat or tallow, instead of the taste of molten butter, which temperature is considerably below that heretofore ordinarily used in rendering fat, and will be found to vary not many degrees above the point already stated.

I also add to the fat while being rendered, for the purpose of aiding in this process, two liters of gastric juice to a hundred (100) kilograms of fat. This gastric juice is made by macerating, for three hours, half of the stomach of a pig or sheep, well washed, and three litres of water containing thirty grams of bi-phosphate of lime. After maceration this macerated substance is passed through a sieve, and then added to the fat under treatment in the proportion of two liters to one hundred (100) kilograms.

The separation of the organized tissues from the fat is aided by the introduction of salt during the rendering; and as soon as there are no lumps of fat visible in the kettle I add about one per cent. of common salt. I stir it for some time. The rendered fat is then allowed to stand until it attains perfect limpidity, when it can be drawn off. By this means the separation is well made, and the organized tissues which do deposit are not altered. I then allow the melted fat to stand in a vessel, maintained at a temperature of about 86° to 98°, until the stearine is crystallized. The mixture of stearine and oleomargarine may then be put in a centrifugal machine; and by the operation of this machine the oleomargarine will pass through the cloth and the stearine remain within; or the mixture may be subjected to pressure in a press. The effect produced in either case is that the oleomargarine practically separates from the stearine and flows out. The oleomargarine thus separated from the stearine, when cooled, constitutes a fatty matter of very good taste, which may replace the butter used in the kitchen. If it is desired, however, to transform it into more perfect butter, I employ the following means: I mix the oleomargarine, as it comes from the press or centrifugal machine, with milk and cream, equal to ten per cent. of the weight of the oleomargarine, the temperature of the milk and cream being about seventy-one (71) degrees, and thoroughly agitate them together. I then let the mixture become completely cold and solid, and then cause it to be worked between rollers, which give it the homogeneousness and the consistency which are the qualities of the natural butter.

The above process of agitating the oleomargarine with milk is intended to be adopted when the butter is to be immediately used. If the butter is intended to be preserved, it will be better to mix the oleomargarine at animal heat with ten per cent. of its weight of water instead of milk or cream, and then agitate the two together, as above described.

I have also found it expedient to mix with the cream or milk, in the first case above described, before agitating, or with the water in the other case above described, before agitating, a fiftieth part of mammary tissue, which is the udder of the cow, minced fine, a one-hundredth part of bicarbonate of soda, and some coloring matter.

It may be desirable to add ordinary butter, and this I do by mixing the oleomargarine and the ordinary butter together at a temperature of about 70° Fahrenheit.

What I claim as my invention, and desire to secure by Letters Patent, is—

(1) The rendering of animal fat at a low temperature, substantially as above set forth, for the production of a fatty matter devoid of disagreeable taste.

(2) As a new product of manufacture, fat rendered at the low temperature, substantially as above described, devoid of disagreeable taste.

(3) The combined process of rendering animal fat at a low temperature and then separating the oleomargarine for the purpose of producing a material adapted to be used as ordinary butter for culinary purposes, or to be further treated for making more perfect butter, substantially as above described.

(4) As a new product of manufacture, oleomargarine obtained from fat rendered at a low temperature and separated from the stearine, substantially as above described.

(5) The agitating of oleomargarine with water or milk for the purpose of making a more perfect imitation of butter, substantially as above described.

(6) The butter-like product produced by the agitation of oleomargarine with water or milk, substantially as above described.

(7) The treatment with artificial gastric juice for facilitating the process of rendering the fat at a low temperature, substantially as above described.

(8) The treatment of the oleomargarine with the mammary tissue of the cow, or mammary pepsin, substantially as above described.

(9) The addition of ordinary butter to oleomargarine, substantially as above described.

H. MÈGE.

Witnesses:
ROBT. M. HOOPER,
M. D. DESHLER.

Garret Cosine took out a patent, February 15, 1876 for "improvement in processes for making artificial butter." From time to time the owners of the Cosine process added to it by patent, but it was finally entirely absorbed by one of the other patents, and now is not recognized as a separate plan.

It is a singular fact that while the French and German governments have encouraged the manufacture and sale of oleomargarine, of course under proper regulations, as a cheap and wholesome substitute for butter, in this country a relentless war has been carried on against it, so that at the present time the manufacture and sale of it are prohibited by laws passed by legislatures and declared constitutional by supreme courts. And this notwithstanding the fact that all scientists who have investigated the subject have pronounced in favor of the wholesomeness of oleomargarine. Prof. Atwater, in a paper on the "Chemistry of Foods," in *The Century*, says in regard to oleomargarine: "This is a case where mechanical invention, aided by science, is enabled to furnish a cheap, wholesome, and nutritious food for the people. Legislation to provide for the official inspection of this, as of other food products, and to insure that it shall be sold for what it is, and not for what it is not, is very desirable. Every reasonable measure to prevent fraud, here as elsewhere, ought to be welcomed. But the attempt to curtail or suppress the production of a cheap and useful food material by law, lest the profits which a class—the producers of butter—have enjoyed from the manufacture of a costlier article may be diminished, is opposed to the interest of a large body of people, to the spirit of our institutions, and to the plainest dictates of justice."

S. P. Sharples, S. B., in a report on the "Adulteration of Food," made to the Massachusetts State Board of Health, says:

"Oleomargarine, when well made, is a fair imitation of genuine butter, being inferior to the best butter, but much superior to the low grades of butter too commonly found in the market. So far as its influence on health is concerned, I can see no objection to it. Its sale as genuine butter is a commercial fraud, and as such very properly condemned by the law. As to its prohibition by law, the same law which prohibited it should also prohibit the sale of lard and tallow, and, more especially, all low-grade butters, which are far more injurious to health than a good, sweet article of oleomargarine. A great deal has been said in regard to the poor grade of fats from which the oleomargarine is made. Any one making such assertions in regard to the fats is simply ignorant of the whole subject. When a fat has become in the least tainted it can no longer be used for the purpose, as it is impossible to remove the odor from the fat after it has once acquired it."

FIG. 258.

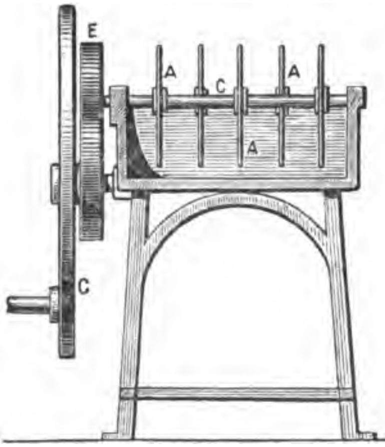
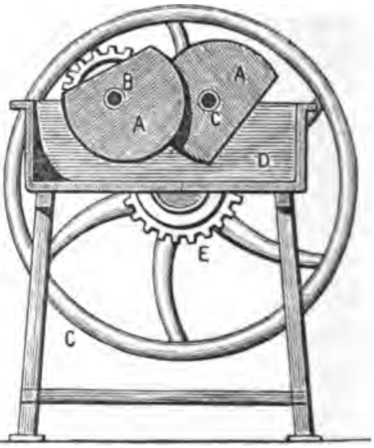


FIG. 259.



HASHING MACHINE.

LONGITUDINAL SECTION. CROSS SECTION.

*A*, knives; *B C*, rolls; *C*, fly-wheel with crank; *D*, trough; *E*, spur-gearing.

A number of other eminent authorities, among them Prof. C. F. Chandler, Prof. Geo. F. Baker, Dr. Henry A. Mott, Jr., Prof.

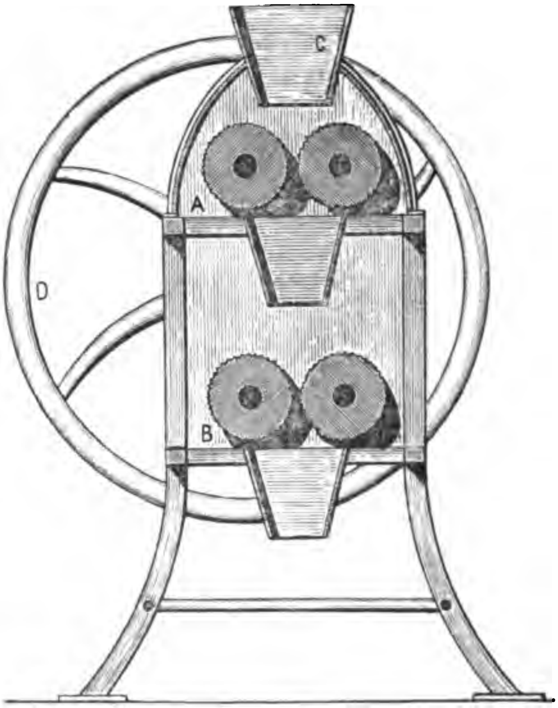
G. C. Caldwell, Prof. S. W. Johnson, Prof. C. A. Goessman and Prof. Henry Morton, have testified to the wholesomeness of artificial butter properly prepared.

Mège, in 1873, prescribed the following method of procedure in the manufacture of artificial butter: 1. Washing, hashing, and melting of the selected raw fat. 2. Crystallization of the rendered fat. 3. Pressing of the crystallized fat. 4. Churning of the oleomargarine.

The fat of best quality from recently killed bullocks is freed from the fleshy parts and thoroughly washed until the water runs off perfectly clear and colorless. It is then cut up in pieces the size of the fist, which are chopped fine in a hashing machine, Figs. 258 and 259. This consists of two rolls, *B C*, provided with a number of sharp knife blades *A*, and is set in motion by the crank *C* and the spur-gearing *E*. The hashed fat can either be rendered at once or, to obtain a greater yield by a complete tearing apart of the fat cells, is brought into a grinding machine, Fig. 260. This consists of two pairs of toothed rolls, *A* and *B*, running at different speeds and set in motion by a crank and spur-gearing (not shown in the illustration).

From these rolls the fragments of fat fall directly into a tank *A* (Fig. 261) heated by steam which is introduced through *B* into a coil upon the bottom of the tank. To each 1000 lbs. of fat are added 300 lbs. of water, ½ lb. of carbonate of potash and 2 stomachs of sheep or pigs. The temperature is then raised to 113° F. After two hours under the influence of the pepsin in the stomachs, the membranes are dissolved and the fat is melted and rises to the top of the mixture. To obtain a product still more closely corresponding to cream-butter, Mège changed the last process by substituting for the potash an addition of acid phosphate of lime and hydrochloric acid, so that a still more artificial gastric juice is formed, and an artificial digestion introduced by heating from 68° to 122° F. When this has acted for about one hour, a yellowish fluid of an agreeable butter-like odor rises to the surface. The fat thus treated by either one of these methods is compounded with about 2 per cent. of common salt and allowed to rest for 2 hours at 113° F. The clear liquid fat is then drawn off through the cock on the rendering tank *A*, Fig. 261, into the cooling tank *C* and cooled to from 68° to 77° F. In 12 to 24 hours the fat is sufficiently granulated and is then cut into pieces. These pieces are wrapped in cotton cloths and put in a hydraulic press. By use of not too strong a pressure in a workroom having a temperature of 77° F., the fat is separated into two nearly equal portions, viz.: stearin 40 to 50 per cent., and fluid oleo 50 to 60 per cent. The stearin remaining in the presses

FIG. 260.



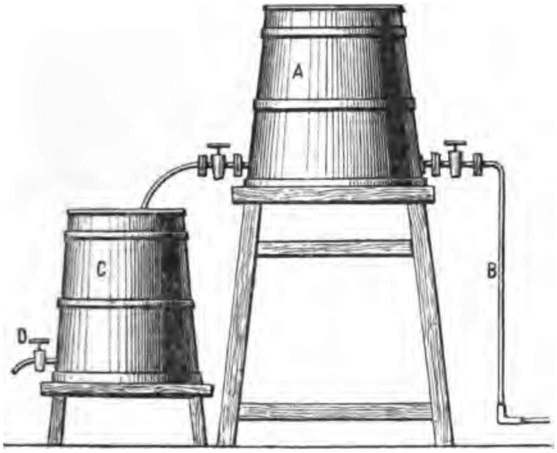
FAT-GRINDING MACHINE.

*A*, upper pair of rolls; *B*, lower pair of rolls; *C*, charging hopper; *D*, fly-wheel with crank.

is used in candle-making; it fuses at 104° to 122° F. The fluid oleo is worked further for household use. When solidified by cooling it has a slightly yellow color and a taste resembling neither that of lard nor of fat, but reminding one rather of rendered butter. In the mouth it liquefies like butter, while beef-fat when taken into the mouth separates into melting oleomargarine, and stearin adhering more or less to the palate.

The oleomargarine thus obtained is made uniform by pressing between cylinders; it is then melted at a low temperature and washed. It forms the fat used for culinary purposes (*graisse de ménage*) or conserving fat (*graisse de conserve*) and is sold in Paris under the name of "*margarine.*"

FIG. 261.



RENDERING AND COOLING VAT.

*A*, rendering vat; *B*, steam-pipe; *C*, cooling vat; *D*, discharge-cock.

To manufacture artificial butter, Mège charges a churn with 66 lbs. of oleomargarine, 25 quarts of cows-milk, which scarcely correspond to 2 lbs. of butter, and 55 lbs. of water which contains the soluble parts of 3½ ozs. of macerated cow's udder in as finely divided a state as possible. For coloring a small quantity of annotto is added. The churn being set in motion, the water and the fat under the influence of the udder pepsin form in a quarter of an hour an emulsion, and appear in the form of a thick paste. By the continued motion of the churn this paste is in a shorter or longer time converted into a butyraceous mass, two hours being, as a rule, sufficient for the purpose. When the operation is finished cold water is introduced into the churn, the object of this being to separate the artificial butter from the buttermilk it retains just like milk butter. For this purpose the product is brought into an apparatus consisting of a kneading machine and two grinding cylinders placed under a rose. The washed artificial butter is of a fine and uniform consistence.

After washing with ordinary water artificial butter retains, according to Boudet's investigation, 12.56 per cent. of water, while milk butter from Isigny* contained 11.94 per cent., and ordinary Calvados* milk-butter 13.28 per cent. According to Boussingault, inferior qualities of milk-butter may contain up to 18 and even 20 and 24 per cent. of water.

The solidifying points of two samples of Mège's butter were determined by Boudet as 71.6° and 62.6° F., while the solidifying point of Isigny butter was found to be 62.8° F., that of ordinary Calvados butter 62.6° F., and that of melted ox fat 89.6° to 91.4° F. The same author also found that the best quality of Isigny butter in a dry state yields 3.13 per cent. casein-like substances insoluble in ether, while Mège's butter leaves behind only 1.20 per cent. of such residues.

The above-mentioned action of mammary tissues (udder of the cow minced fine) on fats used as butter substitutes has been investigated by Tidy and Wagner. By digesting a pure animal fat with chopped up tissues of the udders of cows, the authors found a marked chemical change produced. Oleomargarine or tallow when treated in this way gives rise to both soluble and volatile fatty acids, Since both milk and butter contain a certain amount of mammary tissues, in the form of casts from the mammary glands, it is believed that they also would exert an influence on animal fats. Butter appears to act more vigorously than milk in this way, probably because it contains a larger percentage of mammary tissues.

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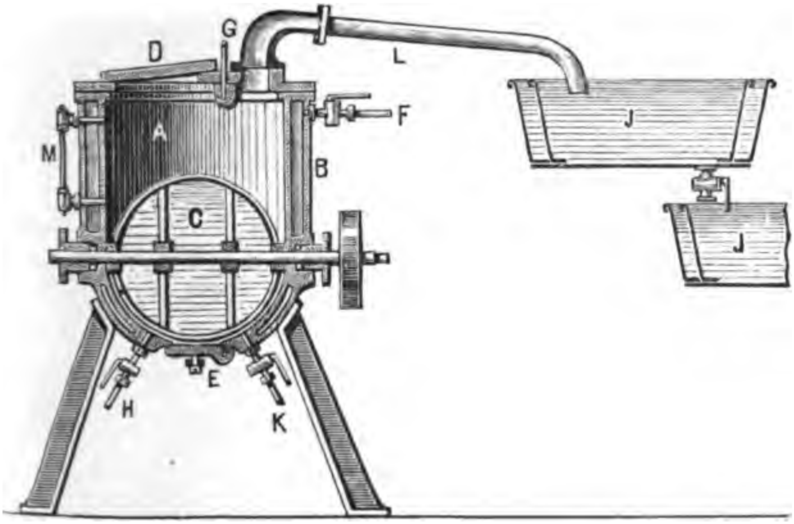
* Celebrated French brands of butter.

In some European factories, especially in Germany, the hashed fat is not treated with stomachs of pigs or sheep, but is directly rendered at a temperature below 122° F., so as to avoid the formation of volatile fatty acids. acrolein and products of decomposition of the membranes, which impart a disagreeable taste to the fat.

FIG. 262.



RENDERING APPARATUS.

*A*, cast-iron caldron; *B*, steam-jacket: *C*, stirring apparatus; *D*, manhole for the introduction of tallow; *E*, opening for empytying the apparatus; *F*, pipe for the introduction of steam; *G*, thermometer; *H*, water-pipe; *J*, settling vessels; *K*, discharge-cock; *L*, pipe; *M*, gauge.

For rendering serves the apparatus shown in Fig. 262. It consists of a cast-iron tank *A*, provided with a steam jacket *B*, and a stirring apparatus *C*. The fat is introduced through *D* and the apparatus emptied through *E*. The pipe *F* conducts steam into the jacket *B*, and a cock (not shown in the illustration, is provided for the discharge of the water of condensation.

When the apparatus is filled about two-thirds full, the manhole *D* is closed, the stirrer *C* set in motion and steam introduced through the pipe *F*. The temperature is controlled by the thermometer *G*. In two hours, when all the fat is rendered, water is admitted through the cock *H* into the tank and the stirring apparatus stopped. The rendered fat separates from the membranes which collect between the water and the fat. The water flowing in forces the fat constantly higher, so that it finally runs through the pipe *L* into the settling vessels *J*, which are also provided with a steam-jacket, so that the fat remains always at the same temperature. When all the fat is discharged from *A* the contents are drawn off through the cock *K*, the manholes are opened, and the caldron is cleansed for a fresh operation.

FIG. 263.

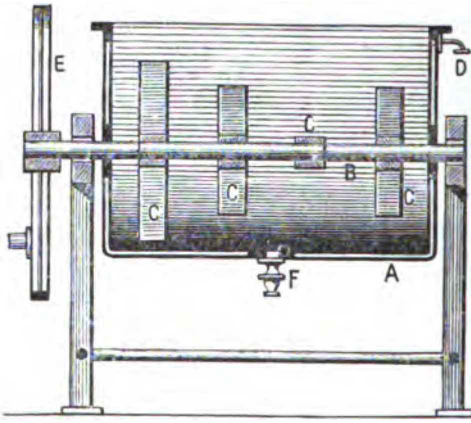
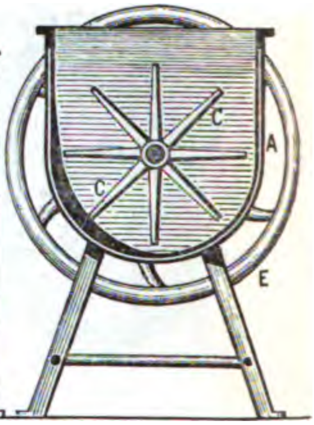


FIG. 264.



CHURN.

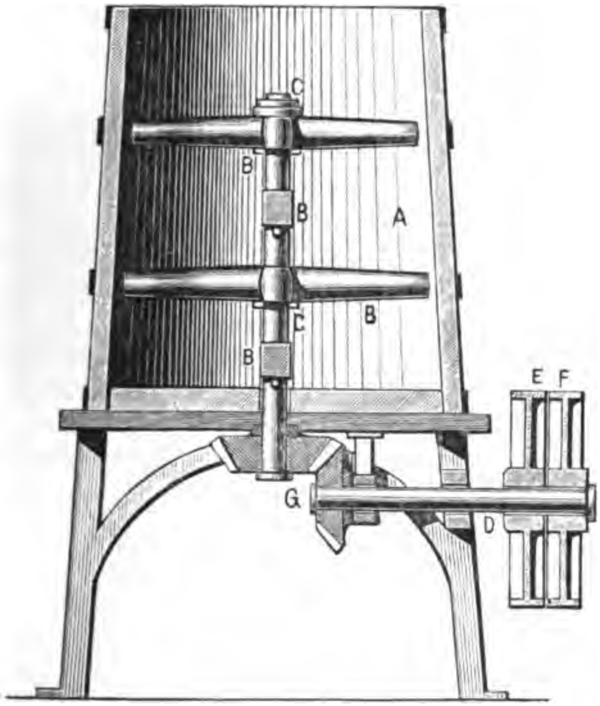
LONGITUDINAL SECTION. CROSS SECTION.

*A*, trough with double walls; *B*, wing shaft; *C*, wings; *D*, conduit for hot water; *E*, fly-wheel with crank; *F*, discharge-cock.

From the settling vessels the fat is drawn off into suitable vessels and removed to the "press-room." Here the melted fat is allowed to remain quietly for about twenty-four hours at a temperature of about 84° F., to allow the oil to granulate by the crystallization of the stearin.

The granulated mass is next filled into bags, set in moulds, and placed on galvanized plates in a series of presses. When these are filled with the bags they are subjected to a gradually increasing pressure, under which the fluid oil is separated, leaving the hard cakes of stearin in the cloths.

FIG. 265.



*A*, vat; *B*, kneading arms; *C*, vertical shaft; *D*, driving shaft; *E F*, pulleys; *G*, bevel gear.

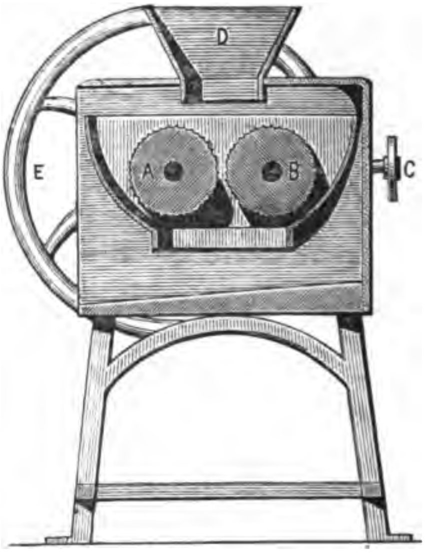
The resulting oily product is a clear, sweet, yellow oil, substantially similar in composition to the oil of butter.

The next step is the conversion of this oil into butter substitute. This is effected by churning with fresh milk in an apparatus resembling ordinary churns. Fig. 263 shows such an apparatus in longitudinal section, and Fig. 264 in cross section.

In large establishments a kneading machine (Fig. 265) driven by machinery is used.

By churning for about twenty minutes the oil is thoroughly emulsionized or broken up into minute globules. A small quantity of anotto is added at this stage to impart a richer color to the product. The emulsionized oil is then drawn off into tubs containing pounded ice, in which it cools suddenly without granulation. Here it remains several hours, when it is thoroughly "worked" by hand and the fragments of ice removed.

FIG. 266.



*A B*, wooden rolls; *C*, small hand-wheel; *D*, charging hopper; *E*, fly-wheel with crank.

To impart the peculiar butter flavor the solidified product is again churned with fresh milk. After this second churning it goes through the same series of operations of working by hand, salting, and packing as ordinary butter. To make the product still firmer and free it from adhering particles of milk and water, it is, in some manufactories, passed through between smooth rolls in an apparatus shown in Fig. 266.

The common method of manufacturing imitation butter in the United States is described by Armsby* as follows:

Although numerous patents have been taken out for the manufacture of imitation butter, and a great variety of materials have been named in the specifications, the process as now conducted is comparatively simple. The raw materials are beef-tallow, leaf-lard, and the best quality of butter, together with small amounts of milk or cream and of butter-color.

From the beef-tallow is prepared the oleomargarine oil of Mège. The caul fat of freshly killed beeves is, after thorough washing first in tepid and then in iced water, allowed to stand in a cold room until thoroughly cold. It is then rendered at a temperature between 130° and 175° F. The resulting oil is allowed to cool slowly until a considerable portion of the stearin and palmitin have crystallized out, and the pasty mass is then subjected to hydraulic pressure. The still fluid portion (about two-thirds of the whole) flows out into a tank of cold water, where it solidifies into a granular mass which is known in the trade as "oleo oil" or simply "oleo." The name "oil" is somewhat misleading, as the product is a granular solid of a slightly yellow color. Fresh leaf-lard treated in substantially the same way as the beef-tallow, yields the "neutral lard" or "neutral" of the trade, also a granular solid of a white color.

The objects of this treatment are two-fold: First, to produce fats as free as possible from taste or odor; second, to remove some of the difficultly fusible stearin and palmitin, in order that the finished product may melt readily in the mouth.

Having thus secured the fats in proper condition, the manufacturer proceeds to mix the "oleo" and "neutral," the proportions varying according to the destination of the product—a warm climate calling for more "oleo," a cold one for more "neutral"—and to flavor the mixture with butter. This flavoring is conducted in large, steam-jacketed vessels provided with revolving paddles, by which their contents can be thoroughly agitated. Here the "oleo" and "neutral" are melted and thoroughly agitated with a certain proportion of milk, or sometimes of cream, and a proper amount of butter-color. Forty-eight gallons of milk per 2,000 pounds of product are stated to be a common proportion. After sufficient agitation, the melted mass is run into cold water, and as it cools is broken up by paddles so as to granulate the mass. After thorough washing, it is salted and worked exactly like butter. The product is known as oleomargarine. Although it contains hardly more than a trace of butter fat, the latter flavors the whole mass so strongly that, when well salted, as it usually is, it might readily pass with an inexpert or careless consumer for a rather flavorless butter. Oleomargarine is the cheapest product made. By adding to the material in the agitator or "churn" more or less pure butter, what is known as butterine is produced, two grades of which are commonly sold, viz., "creamery butterine" containing more, and "dairy butterine" containing less butter.

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* Science, Vol. 7, p. 471.

The method of manufacture used by the firm of Armour & Co., of Chicago, is thus described by Mr. Philip D. Armour*: "The fat is taken from the cattle in the process of slaughtering, and after thoroughly washing is placed in a bath of clean, cold water, and surrounded with ice, where it is allowed to remain until all the animal heat has been removed. It is then cut into small pieces by machinery and cooked at a temperature of about 150° F. until the fat, in liquid form, has separated from the fibrine or tissue, then settled until it is perfectly clear. Then it is drawn into graining vats and allowed to stand a day, when it is ready for the presses. The pressing extracts stearin, leaving the remaining product, which is commercially known as oleo-oil, which, when churned with cream or milk or both and usually a proportion of creamery butter, the whole being properly salted, gives oleomargarine.

"In making butterine neutral lard is used, which is made from selected leaf lard in a very similar manner to oleo-oil, excepting that no stearin is extracted. This neutral lard is cured in salt brine for 48 to 70 hours at an ice-water temperature. It is then taken, and, with the desired proportion of oleo oil and fine butter, is churned with cream and milk, producing an article which, when properly salted and packed, is ready for market.

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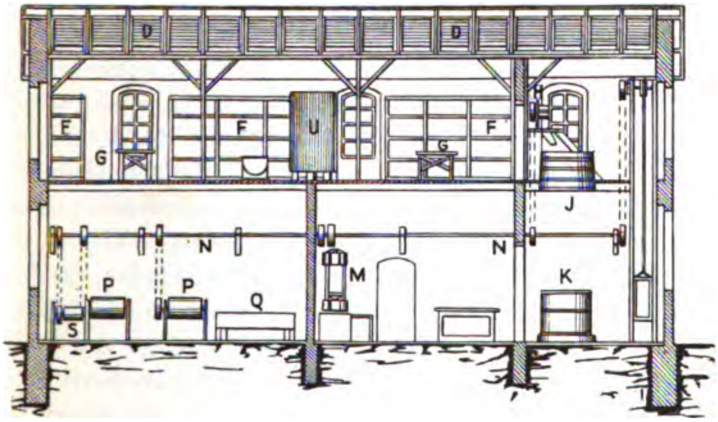
* Senate Mis. Doc. No. 131, Forty-ninth Congress, first session, p. 24.

" In both cases coloring matter is used, which is the same as that used by dairymen to color their butter. At certain seasons of the year, viz., in cold weather, a small quantity of salad oil made from cotton seed is used to soften the texture of the product."

FIG. 267.



ARRANGEMENT OF A PLANT FOR THE MANUFACTURE OF IMITATION BUTTER. LONGITUDINAL SECTION.

The method in use in the manufacture of artificial butter by the firm of Swift & Co. of the town of Lake near Chicago, is according to Gustavus F. Swift* precisely the same as that employed by Armour & Co.

The arrangement of a plant for working 2500 lbs. of fat per day into imitation butter is shown in longitudinal section in Fig. 267.

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* Senate Mis. Doc. No. 131, Forty-ninth Congress, first session, p. 225.

The factory consists of a two-story main building which contains the actual manufacturing rooms and an annex in which are located the steam engine and boiler. The main building is provided with the lantern *D* for ventilating the upper story. In the arrangement of the plant special care has been taken to avoid all unnecessary transport.

The fresh fat when brought into the building is immediately conveyed by an elevator to the upper story and placed upon frames *F*. It is then cleaned from blood, membranes, etc. upon the tables *G*, washed, and hashed in the hashing-machine *H*, the fragments of fat falling through a wooden chute directly into the rendering tank *J*. Upon the bottom of the latter lies a steam-coil. The tank is filled about 1 foot deep with water to which a small quantity of carbonate of potash has been added. Steam being introduced into the coil the mixture of fat and water is heated; the fat rises to the surface, while fibres, etc., fall to the bottom. The rendering operation lasts about 45 minutes, when it is allowed to settle for half an hour, and is then drawn off into the clarifying vat *K*. The latter has double sides and bottom and contains a water-bath which can be heated by means of a steam coil to 104° to 113° F. The fat deposits and is then emptied by means of galvanized buckets into moulds of the same material; the latter are brought into the drying room in which a uniform temperature is maintained by steam-heating. In the drying room the fat solidifies in the moulds and it is then brought into the hydraulic press *M*, which is fed by a pump driven by the main shaft. The oleo-oil running from the presses is caught, and after adding milk, coloring matter, etc., worked in the churn *P*. The mass thus intimately mixed is brought into the trough *Q*, in which it is washed and kneaded until the wash-water runs off clean. After kneading, the butter is brought upon a table and from there into a smoothing apparatus *S*, which in the main consists of a pair of rolls. The butter is then conveyed to the packing room and finally to the cellar, where it is kept until shipped.

All the machinery is driven by means of belts from the main-shaft *N*.

The pure animal fats prepared in the manner described are almost colorless. The tint of genuine butter is imparted to these bodies by various coloring matters, annotto (*Bixa orellana*) being more employed for the purpose than any other substance. Indeed it is used to color genuine butter, which often in winter is almost white in its natural state.

Other coloring matters employed are: Turmeric (*Curcuma longa* and *viridiflora*), saffron (dried stigmas of *Crocus sativus*) and carrot juice (*Daucus carota*). The use of mineral coloring matters like chrome yellow is highly reprehensible from a sanitary point of view.

During the year 1894 the production of oleomargarine in the United States reached 69,622,246 pounds, of which 3,406,058 pounds were exported. This product pays a tax of 2 cents per pound. Illinois produces about two-thirds of the entire quantity, nearly 41,000,000 pounds having been the contribution of that State. Kansas produced nearly 10,000,000; Connecticut and Rhode Island over 6,000,000; Nebraska, 3,475,350, and Ohio over 3,000,000.

In conclusion a table representing the way in which the fatty matter from an ox is utilized in Sarg's artificial butter factory near Vienna is given:

### AN OX YIELDS ABOUT 83 KILOS OF CRUDE TALLOW.

*By carefully separating the fleshy and impure pieces there are obtained:*

A.
28 Kilos of so-called *Kidney-fat.*
After fusing and pressing this yields:

B.
About 55 Kilos crude tallow. This is bleached by melting with steam in the presence of sulphuric acid, and yields:

C.
About 16.5 Kilos of so-called oleomargarine. Washed with water and worked up with milk this yields:

D.
About 8.5 Kilos so-called neutral stearin; fusing point 129.2° F.
Together with E this yields:

F.
About 3 Kilos scraps combined with F¹.

E.
About 41.5 Kilos tallow, fusing point 113° F.
yields together with D:

F¹.
About 13.5 Kilos scraps. This combined with F and pressed to remove the water yields;

G.
About 50 Kilos tallow, fusing point 116° F. This split with water and lime under 10 atmospheres pressure and washed with sulphuric acid yields:

H.
About 47.5° Kilos of crude fatty acids.
These, after cold-pressing, yield:

J.
About 15 Kilos of glycerin of 5° B. which, when evaporated in vacuo, yield:

K.
About 32.5 Kilos of fatty acids.
After hot pressing these yield:

L.
About 15 Kilos fluid fatty acids.
These filtered and crystallized yield:

M.
About 14 Kilos of stearic acid.

N.
About 18.5 Kilos of so-called "returned acid."

O.
About 4 Kilos of solid fatty acid.

P.
About 11 Kilos of oleic acid.

N and O combined and mixed with H yield:

M¹.
About 10 Kilos of stearic acid.

P₁.
About 12.5 Kilos of oleic acid.

J₁.
About 2.5 Kilos of crude glycerin of 30° B.
This after filtering and distilling yields:

C¹.
About 18 Kilos of butter, brought into commerce as oleomargarine, butterine, etc.

M + M¹.
About 24 Kilos of stearic acid, brought into commerce
as: Stearic acid,
" Milly candles (unmixed),
" Stella candles (mixed with paraffin).

P + P¹.
About 23.5 Kilos of oleic acid, brought into commerce
as: Oleic acid,
" Olein soap (soda soap),
" Soft soap (potash soap).

J².
About 2.5 Kilos pure glycerin brought into commerce
as: Refined glycerin,
" Distilled glycerin,
" Glycerin soap,
" Toilet article,
" Roller-mass.

F + F¹.
About 16.5 Kilos of dry scraps used as fertilizer.

### 2. *Cocoa Butter* (*Vegetable Butter*).

Besides oleomargarine, butterine, etc., the appearance and taste of which closely approach genuine butter, a substitute for the latter is at present also prepared from vegetable fat. However, it differs materially in appearance and taste from oleomargarine. Cocoanut oil serves for the basis of this new food-product.

In describing cocoanut oil, p. 105, it has been mentioned that it soon loses its at first peculiar agreeable odor, and by becoming rancid acquires a disagreeable pungent odor and taste.

Regarding the preparation of cocoa butter but little is known, the manufacturers of the product keeping their mode of procedure a profound secret. However, the process is limited to the removal of the coloring matter, of the free fatty acids and of the volatile products possessing a disageeable odor.

For the preparation of cocoa butter, according to Jeserich and Meniert (German patent, No. 19,819), steam of 6 to 8 atmospheres is introduced into melted cocoanut oil, with constant stirring. After two or three hours all the volatile free acids have been removed and the oil is odorless. The admission of steam is then interrupted and to neutralize the non-volatile fatty acids, the fat is mixed, stirring constantly, with about 0.25 per cent. calcined magnesia. After 10 minutes, stirring is interrupted and the fat together with the water condensed from the steam is discharged into a wash-tank filled half-full with warm water and provided with a stirring apparatus. The previously formed magnesia soap together with an excess of magnesia is lifted off from the surface of the fat, the latter washed repeatedly with warm water, and finally after solidifying freed from water by remelting.

Schlinck's method for removing the volatile and odorous fatty acids, etc. from cocoanut oil consists in treatment with alcohol and animal charcoal, whereby a perfectly white mass is obtained, of the consistency of butter and of a sweet, neutral, agreeable flavor.

A method for the preparation of colorless and tasteless cocoanut oil for alimentary purposes has recently been patented by the *Fabriques de Produits Chimiques de Thann et de Mulhouse*, at Thann. We quote from the specifications as follows:

"Many attempts have been made to free cocoanut oil from its peculiar odor and taste and to render it suitable for alimentary purposes, but thus far without being entirely successful.

"It is a well known fact that when steam is introduced into cocoanut oil the odorous constituents are partially removed, but, on the other hand, the oil acquires a somewhat rancid taste. Experiments made to remove this evil show that the odorous constituents may be completely removed by the introduction of superheated steam into the melted oil, observing, however, the precaution of previously expelling the air from the apparatuses by means of a vacuum pump or a current of an indifferent gas, for instance carbon monoxide, and after shutting off the steam, allowing to cool in vacuum or in the atmosphere of the indifferent gas. The process forming the subject of the present invention is carried out accordingly. The cocoanut oil is melted in a suitable vessel and for the expulsion of the air a current of an indifferent gas—carbon monoxide or nitrogen—is introduced, or the air is expelled with the assistance of a vacuum pump or in an otherwise suitable manner. When this has been done to a sufficient extent, superheated steam is introduced until no odor is perceptible. The steam is then shut off and the oil allowed to cool in the current of indifferent gas or in vacuum."

The product brought into market under the name of "cocoa butter" is of a pure white color and of a uniform, salve-like, non-granular nature. It fuses at 80° F. to a perfectly clear fluid which solidifies again at 67° F. The better qualities of cocoa butter have a slight agreeable odor, and melt upon the tongue, exhibiting thereby a mild taste.

An analysis of various samples gave the following values:

| | Fresenius. | Bein. | Rupp. |
|---|---|---|---|
| Fat | 99.979 | 99.756 | 99.993 |
| Water | 0.020 | 0.235 | —— |
| Mineral substances | 0.001 | 0.008 | 0.006 |

The composition of natural butter, oleomargarine and vegetable butter was found to be as follows:

| | Water. | Fat. | Nitrogenous substances. | Milk sugar. | Salts. |
|---|---|---|---|---|---|
| 1. Natural butter | 13.5 | 80.9 | 2.1 | 1.8 | 0.5 |
| 2. Natural butter | 14.6 | 79.5 | 2.8 | 1.9 | 0.5 |
| 3. Oleomargarine | 10.9 | 86.4 | 1.20 | 0.60 | 0.72 |
| 4. Vegetable butter. | 0.235 | 99.756 | —— | — | 0.008 |

# PART II.

## VOLATILE OR ESSENTIAL OILS.

## CHAPTER XIX.

### PROPERTIES OF VOLATILE OILS.

AN extensive class of bodies, nearly all limpid liquids, but some viscid and a few solid, are known under the collective term of volatile or essential oils. They are of such importance for some industries that it would be next to impossible to carry them on without them, examples being the manufacture of perfumery and of liqueurs and cordials. While they form the basis of these industries they are also indispensable in others, such as the manufacture of lacquers and varnishes, and whenever a solvent for resins and certain fats is required.

Most of the volatile oils are of vegetable origin, the animal kingdom furnishing but a few substances, the peculiar odor of which is very likely due to bodies which have to be classed with the volatile oils, though none has thus far been prepared by itself. But one volatile oil—petroleum—is derived from the mineral kingdom. A description of this being not within the scope of this work, we refer our readers who are interested in this vast and interesting subject to the comprehensive work, "Petroleum, its History, Origin, Production, etc," by Wm. T. Brannt. Philadelphia: Henry Carey Baird & Co. 1895.

Before considering the properties of vegetable volatile oils, we will briefly describe the animal substance, referred to above.

*Ambergris*, from *Physeter macrocephalus*, or the sperm whale, has been already described on page 51. On account of its high price it is frequently adulterated. Pure ambergris almost entirely volatizes at a moderate heat, and when burnt leaves very little ash. A small quantity of it, exposed to heat, melts without forming bubbles or scum. It is easily punctured with a heated needle, which, when withdrawn, should come out clean and without anything adhering to it, and the characteristic odor of ambergris should be immediately evolved. The surface should be rugged, the article with a smooth uniform surface being generally factitious. It is much used in perfumery and occasionally in medicine.

*Civet*, an odorous substance of the consistency of honey or butter, obtained from the civet cat (*Viverra civetta*), a carnivorous animal ranking between the weasel and the fox, and inhabiting China, Africa, and the East and West Indies. The civet is secreted in a cavity opening between the anus and the external genitals, and is collected from animals confined for the purpose. It is semi-liquid, unctuous, yellowish, becoming brown and thicker by exposure to the air, of a very strong odor, resembling that of musk, and of a bitter, disagreeable, fatty taste. According to M. Bouthron-Chalard, it contains free ammonia, stearin, olein, mucus, resin, a yellow coloring substance, salts, and a volatile oil, the latter giving the odor to it. It is chiefly used as a perfume.

*Musk* is a peculiar concrete secretion, obtained from *Moschus moschiferus*, an animal bearing a close resemblance to the deer in shape and size, and indigenous to the high plateaus of Asia. The musk is contained in an oval, hairy, projecting sac, found only in the male, situated between the umbilicus and the prepuce. It is from 2 to 3 inches long, and from 1 to 2 broad. In the vigorous male animal the sac contains sometimes 6 drachms of musk, but in the old seldom more than 2 drachms, and none in the young. As soon as the animal is killed, the sac is cut off and dried with its contents, and in this state is sent to market.

Musk is in grains or lumps concreted together, soft and unctuous to the touch, and of a reddish-brown color, resembling that of dried blood. The odor is strong, penetrating, and so

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diffusive that one part of musk communicates its smell to more than 3000 parts of inodorous powder. The taste is bitter, disagreeable, and somewhat acrid.

Musk is very much adulterated, but very little of the genuine article reaching the market. The Chinese are adepts in this sophistication. Dried blood, on account of its resemblance to musk, is among the most common adulterations; but, besides this, sand, iron filings, hair, the dung of birds, wax, asphaltum, and many other substances, are introduced. They are mixed with a small portion of musk, the powerful odor of which is communicated to the entire mass, and renders the discovery of the fraud sometimes difficult. The bags containing the musk should have the characters before described as belonging to the natural sac, and present no sign of having been opened. Genuine musk burns with a white flame, and leaves a white, spongy charcoal; that which burns with difficulty, has a feeble odor and a color either pale or entirely black, feels gritty to the finger, is very moist so as to lose much weight in drying, should be rejected. Musk is used in pharmacy and as a perfume.

*Artificial musk* is obtained, according to Wallmann (German patent No. 231,061) by exposing camphor water to the action of fused chloride of zinc or phosphoric anhydride. The product of reaction is subjected to fractional distillation and the portion passing over between 356° and 445° F. collected. The fluid is rectified over calcium chloride and mixed with equal parts by volume of amyl alcohol. The mixture is introduced, a little at a time, into five times its volume of Nordhausen (fuming) sulphuric acid and 7 volumes of 66° ordinary sulphuric acid. After agitating three or four hours the mixture is diluted with water, neutralized with carbonate of soda and again mixed with amyl alcohol. The amylic solution is separated, agitated with lead acetate solution and the separated portion of the fluid evaporated at a gentle heat. The amorphous residue thus obtained is treated with 12 parts of a mixture of equal parts of fuming nitric acid and 66° sulphuric acid, heated for three hours at 176° F., and washed in a large quantity of cold water. The precipitate obtained is dissolved in alcohol and recrystallized; it evolves a strong odor of musk.

*Castor*, or *castoreum*, a peculiar concrete substance obtained from *Castor fiber*, or the beaver. In both sexes between the anus and external genitals are two pairs of membranous follicles, of whicn the lower and larger are pear shaped, and contain an oily, viscid, highly odorous substance analogous to civet and musk, which is called castor. After the death of the animal, the follicles containing it are removed and dried, either by smoke or in the sun, and in this state are sent to market. Good castor has a strong, fetid, peculiar odor, a bitter and nauseous taste, and a color more or less tinged with red. F. Woehler has ascertained the existence of silicin in castor; also that it contains a small quantity of carbolic acid, to which he ascribes its odor.

*The volatile oils derived from the vegetable kingdom* are sometimes designated as "distilled oils," from the mode in which they are usually procured, and "essential oils," because they posses in a concentrated form the odor and properties of the plants or vegetable substances whence they are obtained. Their presence confers upon flowers, leaves, fruits, seeds, roots, barks and woods their peculiar and characteristic odor, and to them we are indebted for our most delightful perfumes and our choicest spices and aromatics. The odors exhaled by our own domestic plants have been but little studied, but the southern as well as many northern districts of the United States are well adapted for the cultivation of quite a number of species of plants which might be made to yield highly valuable articles of commerce. Among the plants which might furnish oils for the perfumer's use are, for instance, the wall-flower, the lily, lilac, and mignonette.

We would here call attention to the frequent supposition that a plant is the richer in oil the stronger its odor. If this were the case, the hyacinth, for instance, would contain more oil than the coniferæ, but, in fact, it contains so little that it can be separated only with the greatest difficulty. The odor does not depend on the quantity, but on the quality of the oil; a plant may diffuse but little odor and still contain much volatile oil.

In many plants, for instance, the true *coniferæ*, volatile oil is found in every part, while in others it is contained in special cells, glands, cavities, or canals within the plants, either in a separate condition or intermixed with resinous substances, the mixtures, in the latter case, forming oleo-resins, balsams, or resins, according as the product is viscid or solid and hard.

In many plants the oil is exclusively found in the flowers, as in the rose, violet and many others; in others in the fruit, as in nutmegs; and in some most abundantly in the expanded buds, as in the clove.

In many *lauraceæ* the greater quantity of oil occurs in the bark, as in *Cinnamomum zeylonicum*, though some is found also in the flowers and fruits.

In fragrant fruits, as the lemon and orange, the oil occurs only in the outer rind, the pulp containing not a trace. In some other plants the oil is stored in the roots while the plant itself does not contain a particle.

Many plants are provided with peculiar glands containing an abundance of volatile oil. We would here only mention a species of dittany (*Dictamnus albus*), a beautiful plant of Southern Germany, the stem of which is entirely covered with oil glands, diffusing an agreeable lemon-like odor. Country folks state that the plant is so rich in oil that on a hot summer's night the vapor exhaled will ignite by bringing a lighted candle near a colony of these plants; though having frequently tried the experiment we have never succeeded in doing this.

Sometimes the volatile oil is contained only in certain parts of the fruit, as, for instance, in that of the hop, which secretes a fine, pulverulent body called "lupulin," containing besides other substances a considerable quantity of oil.

It would seem that the volatile oils of many plants do not participate in the actual living process of the plant itself; we find them inclosed in special oil sacs or stored in large quantities between the vascular bundles of the wood, camphor being an example.

There are several combinations which from their properties must be classed with the volatile oils. They do not exist ready formed in the plants whence they are obtained, but result from chemical change of inodorous principles, oil of bitter almonds being an example of this class. It does not pre-exist in the almond, but is produced by the reaction of water upon a glycoside called "amygdalin" contained in it, through the intervention of another constituent called "emulsin." Amygdalin exists also in the leaves of the common laurel, in those of the bird cherry (*Prunus padus*), in the sprouts and bark of the mountain ash, and in other plants belonging to the family of *Drupaceæ* and *Amygdaleæ*.

The volatile oils vary so much in their physical as well as their chemical properties that they can be brought into one group only by an arbitrary method. While most of them are colorless and fluid, we find all imaginable differences in their aggregate condition; many are very limpid, others appear as solid bodies of crystalline structure, and between these two extremes occur all possible intermediate grades.

*Solubility of volatile oils.* Most volatile oils are insoluble, or only with difficulty and sparingly soluble, in water, but in alcohol, ether, chloroform, benzine, carbon disulphide, fat oils, etc., they dissolve freely, though, of course, not with the same facility in all of them. Of special interest and of technical importance is the behavior of volatile oils towards water and alcohol.

*Behavior towards water.* For pharmaceutical purposes it is frequently of importance to divide volatile oil as finely as possible in water (which might be called "dissolving"). Various methods have been proposed for this purpose. L. Igel pours the oil upon filtering paper, cuts the latter into small pieces and shakes them with water (1 gramme water to 1 drop oil) until the whole forms a paste. He then adds carefully, with constant agitation, enough water to allow of filtering. Q. Curtmann rejects the customary method of finely dividing the volatile oil by triturating with magnesia or phosphate of lime, and then taking it up with water, but recommends for the purpose talc or infusorial earth previously boiled with hydrochloric acid. Finally Bergmann draws attention to the fact that water containing ammoniacal soap is capable of absorbing large quantities of volatile oil.

*Behavior towards alcohol.* According to their solubility in alcohol, the volatile oils may be grouped as follows:

1. *Oils not soluble in every proportion in absolute alcohol:* Oils of arnica, cubebs and rose.

2. *Oils soluble in every proportion in alcohol of 0.85 specific gravity:* Oils of asafœtida, bitter almond, cajaput, calamus, caraway, cardamon, cinnamon, cloves, crisped (curled) mint, elemi, galangal, garlic, lavender, marjoram, massoy, mustard, rosemary, sage, spike-lavender, spiræa, tansy, thuja, thyme, valerian root, wild ginger (sarabacca), wild thyme, wintergreen, wormseed (American), wormseed (Levant), wormwood.

3. *Oils soluble in 0.5 to 6 parts of alcohol of 0.85 specific gravity:* Oils of aniseed (5), arnica root (5 to 6), balm (5 to 6), bergamot (0.5), cascarilla (1 to 2), cumin (3), fennel (2 to 4), hyssop (1 to 4), mace (6), neroli (1 to 3), origanum (6), parsley (1 to 3), peppermint (1 to 3), rue (1), sassafras (4 to 5), savin (2), star-anise (5 to 6), yarrow or milfoil (1).

4. *Oils soluble with difficulty in alcohol of 0.85 specific gravity;* Oils of chamomile (8 to 10), fennel flower (30), sweet gale (50), turpentine (10 to 12).

5. *Oils dissolving with difficulty and not clear in alcohol of 0.85 specific gravity:* Oils of arnica flowers (100), copaiba (20 to 30), cubebs (27), juniper-berry (10 to 12), lemon (10), orange peel (7 to 10), rose (100).

*Oils which do not cause turbidity.* For the manufacture of liqueurs and similar purposes, it is of importance that the volatile oils used for imparting odor and taste should not yield turbid secretions when mixed with alcohol. Oxygenated volatile oils contain fewer substances causing turbidity than oils free from oxygen, and oils dissolving with difficulty in alcohol more than oils dissolving with greater ease. To the oils which do not cause turbidity belong those enumerated in the second, and perhaps in the third, group, while group 4 and especially groups 1 and 5 embrace oils which cause much turbidity. In the fractional distillation of volatile oils a heavier portion is finally obtained, which in commerce is known as *extra strong oil.* Such oil, as a rule, dissolves clear in 50 per cent. alcohol, and even does not make turbid 25 per cent. whisky; hence it belongs to the oils which do not cause turbidity.

Volatile oils ignite with great ease and burn with a fierce, smoky flame, depositing a large amount of carbon. They differ from the fat oils in many important respects; they are not unctuous to the touch, but rather make the skin brittle and rough; they leave no permanent grease stain, distil at various temperatures unchanged, have a powerful odor, some agreeable and others repulsive, and a hot, burning taste, and in chemical constitution have no relationship to the fats and oils.

Many of them are not actual chemical combinations, in so far as we understand by that term a thoroughly homogeneous body, but are unstable mixtures consisting of at least two bodies differing from each other. The behavior of many volatile oils under an increase of temperature gives us an insight into these relations. Such oils, which consist very likely of but one combination, have a boiling point remaining almost always constant, while that of those consisting of a mixture of combinations varies considerably, this variation depending very likely on the greater or smaller quantity of one of the combinations.

The oils behave in a similar manner under a decrease of temperature. At certain temperatures the simple combinations solidify throughout their entire mass, while those consisting of a mixture of two combinations, on exposure to low temperatures, separate into two portions, one solid, called "stearoptene," and the other liquid, called "elæoptene." This behavior is sometimes utilized as a means of testing the purity of volatile oils.

It would seem that all fresh volatile oils in a state of absolute purity are colorless, there being no reason to believe that the brown, red, green, blue, or other coloration frequently found depends on foreign matters dissolved in the oil. The blue or green color exhibited by certain volatile oils, chiefly from plants of the composite order, wormwood, chamomile, for example, is due to an oily compound, *cærulein*, $C_{20}H_{10}O_7$, which may be separated by distillation, passing over with the last portion of the oil, but has not been obtained pure. The solution of this blue substance in oils or alcohol exhibits, when examined by a hollow wedge and prism, a very characteristic spectrum, in which the orange and yellow rays are first absorbed, and at a certain depth nothing is suffered to pass except the greenish-blue rays and the extreme red band.

The volatile oils are all powerfully acted on by oxygen, which affects their color, consistency, odor, and constitution. In specific gravity they range from about 0.750 to 1.170, most of them being specifically lighter than water.

Most bodies, under otherwise equal conditions, show always exactly the same specific gravity, the variations being so slight that they can be justly ascribed to errors of observation. One and the same volatile oil frequently shows, however, such variation in specific gravity that we are forced to ascribe it to the constitution of the oil itself. And such, no doubt, is the case, as these variations are chiefly observed in oils separating, on exposure to cold, into stearoptene and elaeoptene. By separating the former as much as possible from the latter, the specific gravity of each separate body varies but little.

Considerable variations in specific gravity occur, however, also in oils not considered mixtures, but they belong to the very unstable bodies, so that an old oil possesses entirely different properties from the same oil recently prepared. It would, therefore, seem that for the exact determination of the specific gravity of a volatile oil it should be subjected to examination immediately after its preparation from the plant or vegetable substance, which should be as fresh as possible.

The annexed table of specific gravities has the advantage of having been prepared in the factory of Messrs. Schimmel & Co., which is the largest in Germany, and can, therefore, be relied upon:

| Oil of | Specific gravity at ° F. | | | Remarks. |
|---|---|---|---|---|
| | 50° | 59° | 68° | |
| Angelica | 0.860 | 0.858 | 0.853 | |
| Aniseed | — | 0.985 | 0.980 | |
| Bergamot, 1st quality | 0.887 | 0.883 | 0.880 | Reggio. |
| Bitter almond | 1.063 | 1.060 | 1.055 | average 1.062. |
| Cajeput | 0.927 | 0.925 | 0.922 | green. |
| Calamus | 0.961 | 0.959 | 0.957 | |
| Caraway, double rectified | 0.911 | 0.908 | 0.905 | from Dutch caraway. |
| Caraway, double rectified | 0.905 | 0.900 | 0.986 | meadow caraway. |
| Cardamon | 0.902 | 0.900 | 0.897 | Ceylon. |
| Cassia | 1.073 | 1.068 | 1.063 | = cinnamon flower oil. |
| Cassia, rectified | 1.058 | 1.055 | 1.052 | average 1.05 to 1.06. |
| Cedar wood | 0.948 | 0.945 | 0.940 | |
| Cinnamon (Ceylon) | 1.035 | 1.030 | 1.027 | average 1.03 to 1.035. |
| Citronella | 0.900 | 0.896 | 0.893 | |
| Clove | 1.065 | 1.062 | 1.059 | average 1.060 to 1.065. |
| Clove stalks | 1.065 | 1.061 | 1.057 | average 1.060 to 1.065. |
| Coriander | 0.872 | 0.867 | 0.864 | average 0.860 to 0.870. |
| Crisped mint | 0.930 | 0.925 | 0.922 | German, rectified. |
| Cubebs | 0.918 | 0.915 | 0.912 | |
| Cumin | 0.925 | 0.922 | 0.918 | |
| Dill | 0.905 | 0.900 | 0.896 | |
| Eucalyptus | 0.925 | 0.922 | 0.918 | from E. globulus. |
| Fennel, 1st quality | 0.975 | 0.970 | 0.965 | from seed, rectified. |
| Ginger | 0.885 | 0.882 | 0.878 | |
| Juniper berry | 0.863 | 0.858 | 0.855 | double rectified. |
| Lemon | 0.856 | 0.854 | 0.851 | |
| Mace | 0.858 | 0.855 | 0.852 | |
| Mustard | 1.030 | 1.025 | 1.020 | |
| Orange | 0.854 | 0.850 | — | sweet. |
| Peppermint | 0.906 | 0.903 | 0.901 | average 0.900 to 0.910; brand F. S. & Co., New York. |
| Peppermint | 0.905 | 0.900 | 0.898 | Mitcham; average 0.900 to 0.905. |
| Sandalwood | 0.978 | 0.975 | 0.973 | superfine East Indian. |
| Sassafras | 1.068 | 1.065 | 1.060 | average 1.05 to 1.07. |
| Star anise | 0.990 | 0.985 | 0.980 | |
| Valerian root | 0.947 | 0.945 | 0.940 | |
| Wintergreen | 1.189 | 1.185 | 1.182 | natural. |

*Optical behavior of volatile oils.*—Volatile oils possess great refractive power and exhibit great diversity in their action on polarized light, some turning the plane of polarization to the left and some to the right, while others are entirely inactive. But for practical purposes this test is of but little value, being difficult to execute and requiring complicated apparatus. And besides, as shown by Flückiger's practical notes upon the rotatory powers of volatile oils, the polaristrobometer does not give trustworthy results, since one and the same oil will exercise a different action upon the ray of polarized light, when in a fresh condition, to what it does when it has become old. Moreover, a mixture of low-priced right and left rotatory oils can be prepared which, being optically inactive, cannot be detected as an adulterant by the polaristrobometer. Further, differences of soil and temperature, as well as the time of the collection of the plants, are often answerable for variations in the rotation by the oil.

The following table shows the rotatory power of some oils, together with their refractive indices for the lines A, D, and H, as determined by Gladstone,* and also their specific gravities. The rotatory power was determined for a column of liquid ten inches long:

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* Jour. of Chem. Soc., XVII., 3.

| Crude oils. | Specific gravity at 15.5° C. | Refractive indices. | | | | Rotation. |
|---|---|---|---|---|---|---|
| | | Temp. | *A.* | *D.* | *H.* | |
| Anise | 0.9852 | 16.5° | 1.5433 | 1.5566 | 1.6118 | —1° |
| Bay | 0.8808 | 18.5 | 1.4944 | 1.5022 | 1.5420 | —6 |
| Bergamot | 0.8825 | 22.0 | 1.4559 | 1.4625 | 1.4779 G. | +23 |
| " Florence | 0.8804 | 26.5 | 1.4547 | 1.4614 | 1.4760 G. | +40 |
| Birch bark | 0.9005 | 8.0 | 1.4851 | 1.4921 | 1.5172 | +38 |
| Cajeput | 0.9203 | 25.5 | 1.4561 | 1.4611 | 1.4778 | 0 |
| Calamus | 0.9388 | 10.0 | 1.4965 | 1.5031 | 1.5204 G. | +43.5 |
| Caraway | 0.8845 | 19.0 | 1.4601 | 1.4671 | 1.4886 | +63 |
| " Hamburg 1st dist. | 0.9121 | 10.0 | 1.4829 | 1.4903 | 1.5142 | |
| " " 2d " | 0.8832 | 10.5 | — | 1.4784 | — | |
| Cascarilla | 0.8956 | 10.0 | 1.4844 | 1.4918 | 1.5158 | +26 |
| Cassia | 1.0297 | 19.5 | 1.5602 | 1.5748 | 1.6243 G. | 0 |
| Cedar | 0.9622 | 23.0 | 1.4978 | 1.5035 | 1.5238 | +3 |
| Cedrat | 0.8584 | 18.0 | 1.4671 | 1.4731 | 1.4952 | +156 |
| Citronella | 0.8908 | 21.0 | 1.4599 | 1.4659 | 1.4866 | —4 |
| " Penang | 0.8847 | 15.5 | 1.4604 | 1.4665 | 1.4875 | —1 |
| Cloves | 1.0475 | 17.0 | 1.5213 | 1.5312 | 1.5666 | —4 |
| Coriander | 0.8775 | 10.0 | 1.4592 | 1.4652 | 1.4805 G. | +21 ? |
| Cubebs | 0.9414 | 10.0 | 1.4953 | 1.5011 | 1.5160 G. | |
| Dill | 0.8922 | 11.5 | 1.4764 | 1.4834 | 1.5072 | +206 |
| Elder | 0.8584 | 8.5 | 1.4686 | 1.4749 | 1.4965 | +14.5 |
| Eucalyptus amygdalina | 0.8812 | 13.5 | 1.4717 | 1.4788 | 1.5021 | —136 |
| " oleosa | 0.9322 | 13.5 | 1.4661 | 1.4718 | 1.4909 | +4 |
| Indian geranium | 0.9043 | 21.5 | 1.4653 | 1.4714 | 1.4868 G. | —4 |
| Lavender | 0.8903 | 20.0 | 1.4586 | 1.4648 | 1.4862 | —20 |
| Lemon | 0.8498 | 16.5 | 1.4667 | 1.4727 | 1.4946 | +164 |
| Lemon-grass | 0.8932 | 24.0 | — | 1.4705 | — | —3 ? |
| " Penang | 0.8766 | 13.5 | 1.4756 | 1.4837 | 1.5042 | 0 |
| Mint | 0.9342 | 19.0 | 1.4767 | 1.4840 | 1.5015 G. | —116 |
| " | 0.9105 | 14.5 | 1.4756 | 1.4822 | 1.5037 | —13 |
| Myrtle | 0.8911 | 14.0 | 1.4623 | 1.4680 | 1.4879 | +21 |
| Myrrh | 1.0189 | 7.5 | 1.5196 | 1.5278 | 1.5472 G. | —136 |
| Neroli | 0.8789 | 18.0 | 1.4614 | 1.4676 | 1.4835 G. | +15 |
| " | 0.8743 | 10.0 | 1.4673 | 1.4741 | 1.4831 F. | +28 |
| Nutmeg | 0.8826 | 24.0 | 1.4644 | 1.4709 | 1.4934 | +44 |
| " Penang | 0.9069 | 16.0 | 1.4749 | 1.4818 | 1.5053 | +9 |
| Orange peel | 0.8509 | 20.0 | 1.4633 | 1.4699 | 1.4916 | +32 ? |
| " " Florence | 0.8864 | 20.0 | 1.4707 | 1.4774 | 1.4980 | +216 |
| Parsley | 0.9926 | 8.5 | 1.5068 | 1.5162 | 1.5417 G. | —9 |
| Patchouli | 0.9554 | 21.0 | 1.4990 | 1.5050 | 1.5194 G. | |
| " Penang | 0.9592 | 21.0 | 1.4980 | 1.5040 | 1.5183 G. | —120 |
| " French | 1.0119 | 14.0 | 1.5074 | 1.5132 | 1.5202 F. | |
| Peppermint | 0.9028 | 14.5 | 1.4612 | 1.4670 | 1.4854 | —72 |
| Petit-grain | 0.8765 | 21.0 | 1.4536 | 1.4600 | 1.4808 | +26 |
| Rose | 0.8912 | 25.0 | 1.4567 | 1.4627 | 1.4835 | —7 |
| Rosemary | 0.9080 | 16.5 | 1.4632 | 1.4688 | 1.4867 | +17 |
| Rosewood | 0.9064 | 17.0 | 1.4843 | 1.4903 | 1.5113 | —16 |
| Sandalwood | 0.9750 | 24.0 | 1.4959 | 1.5021 | 1.5227 | —50 |
| Thyme | 0.8843 | 19.0 | 1.4695 | 1.4754 | 1.4909 G. | |
| Turpentine | 0.8727 | 13.0 | 1.4672 | 1.4732 | 1.4938 | —79 |
| Verbena | 0.8812 | 20.0 | 1.4791 | 1.4870 | 1.5059 G. | —6 |
| Wintergreen | 1.1423 | 15.0 | 1.5163 | 1.5278 | 1.5737 | +3 |
| Wormwood | 0.9122 | 18.0 | 1.4631 | 1.4688 | 1.4756 F. | |

*Spectroscopic examination of volatile oils.* W. N. Hartley and A. K. Huntington found the terpenes, $C_{10}H_{16}$, capable of absorbing quite vigorously the ultra violet rays of the spectrum, while the sesquiterpenes, $C_{15}H_{24}$, possess a much greater absorbent power for the rays of greater refrangibility, and cymol, $C_{10}H_{14}$, furnishes such a characteristic absorption-spectrum that with its assistance it can be detected in a dilution with 5,000 volumes alcohol. Benzol and its derivatives absorb the ultra violet much more vigorously than terpenes; by diluting the volatile oils with alcohol such bodies of the aromatic series can frequently be recognized with certainty, and even their quantity be estimated.

The spectroscopic behavior may serve as a basis for the following division of the volatile oils:

1. *Oils which furnish continuous spectra:* Australene from oil of turpentine; birch bark oil; cajeputene and carvene; oils of calamus, cedar wood, citronella, cubebs and lemon; hesperidin from orange peel oil; oils of lemon grass, juniper, lavender, linalol; menthol and the hydrocarbon from nutmeg oil; oils of patchouli, rose, rosewood, rosemary, sandal wood and vetiver; terebene and terebenthene.

2. *Oils which exhibit the absorption-spectrum of cymol:* Oils of thyme, lemon, nutmeg and caraway.

3. *Oils whose dilute solutions yield absorption-bands:* Oils of aniseed, bay, bergamot, bitter almonds, cassia, peppermint, cloves, pimento and thyme; caravol from caraway oil, myristicol from nutmeg oil, and the blue oil of patchouli oil.

C. Hock has especially examined oils which contain cœrulein. Oils of chamomile, wormwood and yarrow, or milfoil, give in red and orange, at B, C, and C $\frac{2}{3}$ D, exactly in the same position three absorption-bands, the pure blue oil exhibiting them still more strongly. According to Tischomirow, the oils of bergamot and cajeput give the chlorophyl spectrum. On the other hand, the oils of orange flowers and peppermint give absorption-spectra only when subjected to special chemical treatment, by shaking the former with a saturated solution of bisulphate of soda, and the latter with nitric or acetic acid. Oil of orange flowers becomes thereby red to purple, and peppermint oil green to blue; both showing then the characteristic absorption of light.

*Chemical properties and composition of volatile oils.* In chemical constitution the volatile oils vary very much, but they are invariably rich in carbon. The greater number consists of carbon and hydrogen only; another group of carbon, hydrogen and oxygen; and a third class, limited in number, of carbon, hydrogen, oxygen and sulphur.

By taking their chemical composition as a basis we may divide the volatile oils into three principal divisions:—

1. Volatile oils free from oxygen, or hydrocarbons consisting of CH.
2. Oxygenated volatile oils consisting of CHO.
3. Volatile oils containing sulphur and consisting of CHOS.

Of the hydrocarbons, which constitute the principal proportion of volatile oils, terpene, $C_{10}H_{16}$, is the most important. Terpene is the chief constituent of the various kinds of oil of turpentine, and a hydrocarbon of precisely the same composition is contained in the oils of bergamot, orange, and other species of *Citrus*, and in a great number of other essential oils. But although agreeing in chemical formula, these terpenes vary very much as regards physical properties, the different compounds having different boiling points, specific gravities, etc. Their odor also varies much, and most of them are optically active, some turning the plane of polarization to the right, others to the left. The cause of this physical isomerism is not yet known. Under the name terpene are also included two series of hydrocarbons, polymeric with the $C_{10}H_{16}$ series, having, respectively, the formula $C_{15}H_{24}$ and $C_{20}H_{31}$. The former of these, $C_{15}H_{24}$, is of comparatively frequent occurrence, being found in the volatile oils of cloves, cubebs, patchouli, and several others.

The terpenes of the formula $C_{10}H_{16}$, by their oxidation in presence of water, give off indirectly peroxide of hydrogen, $H_2O_2$, and yield cymene $C_{10}H_{14}$, a hydrocarbon found naturally in the volatile oils of cumin, thyme, and some others. The property of evolving peroxide of hydrogen, possessed by cymene in common with terpene, explains the well-known oxidizing and antiseptic influence of common turpentine oil. The oxygenated bodies in volatile oils, when formed by direct oxidation of the hydrocarbons, generally have feebly acid properties developing through viscid oleo-resins and balsams into solid resins.

The oxygenated compounds are very varied in their constitution, some being of the nature of acids or aldehydes, while others exhibit the characters of alcohols or ethers. The most characteristic oxygenated compounds in volatile oils are camphors, the type of which is common, or Japan camphor, $C_{10}H_{16}O$. Compounds isomeric with common camphor occur in wormwood, mint, and other labiate plants, and in chamomile and galbanum. Camphors isomeric with Borneo camphor, $C_{10}H_{18}O$, are found in coriander oil, cajeput oil, and Indian geranium oil. Isomeric camphors having the formula $C_{10}H_{20}O$ form the menthol of peppermint and the eucalyptol of *Eucalyptus globulus*, and patchouli camphor has the composition $C_{15}H_{26}O$. The other oxygenated bodies in crude volatile oils mostly ally themselves with compounds of the aromatic series, so called on account of the large proportion of its members that are represented by the natural products obtained in volatile oils, balsams and resins.

The essential oils, which contain sulphur, of which the allyl sulphide, $C_6H_{10}S$, or oil of garlic, is a type, have generally a pungent, penetrating, and disagreeable odor.

*Constituents of volatile oils.* The separate classes of combinations here to be discussed are: Hydrocarbons, especially terpenes; alcohols; phenols; aldehydes; acids; ethers; nitrils; sulphides; isosulphocyanates, etc.

*Hydrocarbons.* Under hydrocarbons are understood compounds of carbon and hydrogen. Their number is exceedingly large, this being rendered possible by the peculiarity of carbon to combine to groups of very different values.

In volatile oils the following hydrocarbons occur: Heptane, $C_7H_{16}$; hexadecane, $C_{16}H_{34}$; cymol, $C_{10}H_{14}$; terpene, $C_{10}H_{16}$ (pinene, limonene, dipentene, phellandrin, sylvestrene); sesquiterpene, $C_{15}H_{24}$; diterpene, $C_{20}H_{32}$; polyterpene, $(C_5H_8)_4 + x$.

*Heptane*, $C_7H_{16}$, occurs in the resin of *Pinus Sabiniana* Douglas, the nut pine or digger pine of California, and is obtained from it by distillation. Commercial name: *Abietene.* It is an almost colorless, mobile fluid, of an orange-like odor. When pure it boils at 213.8° F. Specific gravity at 32° F., 0.7.

*Hexadecane*, $C_{16}H_{34}$. A solid body fusing at 69.8° F. and boiling at 532.4° F. It forms laminæ with a nacreous lustre. It is probably a constituent of the stearoptene of rose oil, which, in an impure state, fuses at 90.5° F. and boils at 521.6° F.

*Styrolene*, $C_6H_5C_2H_3 = C_8H_8$. Occurs in storax oil. It is a mobile, very refractive and fragrant liquid, boiling at 292° F. Specific gravity = 0.925 at 32° F.

*Cymol*, $C_{10}H_{14}$ is an aromatic hydrocarbon, viz., methyl-propyl-benzol. It occurs in Roman cumin oil, in the oil of the water hemlock, in the oils of thyme, ajowan, summer savory, wild thyme, eucalyptus, and even in old oil of turpentine. It is a colorless, very refractive liquid, possessing an aromatic odor and boiling at 347° F. Specific gravity = 0.791 at 32° F. It has a characteristic absorption-spectrum.

*Terpenes.* All terpenes possess the common formula $C_{10}H_{16}$; they are aromatic hydrocarbons and stand in relation to cymol.

*Pinene*, $C_{10}H_{16}$ is the essential constituent of oils of turpentine pine needles, juniper berries, sage, rosemary, eucalyptus, mace, etc. It occurs in three optically isomeric combinations, namely, *levo-pinene or terbethene*, *dextro-pinene or australene* and *inactive pinene.* Pinene is a colorless liquid of a slightly aromatic odor, boiling at 311° to 312.8° F. Specific gravity = 0.8707 at 32° F., and 0.858 at 68° F. By heat and acids it is converted into other terpenes. Of its derivatives may be mentioned: Liquid dibromide, $C_{10}H_{16}Br_2$; liquid dichloride, $C_{10}H_{16}Cl_2$; solid monohydrochloride, $C_{10}H_{16}HCl$ (the so-called artificial camphor) which fuses at 257° F. and boils at 406.4° F., solid nitrosochloride, $C_{10}H_{16}NOCl$, which fuses at 217.4° F., and with alcoholic lye is converted into $C_{10}H_{14}NOH$, fusing at 264.2° F.; solid nitrosobromide, $C_{10}H_{16}NOBr$, fusing at 195.8° to 107.6° F. Terebenthene occurs, for instance, in French oil of turpentine, aniseed oil and thyme oil; australene in American and Russian oils of turpentine, wormwood oil and spearmint oil.

*Camphene*, $C_{10}H_{16}$, seems to occur in oils of myrtle and rosemary. It is, however, chiefly a product of the conversion of other terpenes and terpene derivatives. It is a solid paraffin-like terpene, fusing at 118.4° to 120.2° F., and distilling at 320° to 321.8° F. It dissolves with difficulty in alcohol, and may be obtained from the alcoholic solution in crystals. It yields monobromocamphor, $C_{10}H_{15}Br$, an oily liquid boiling at 446° to 464° F. When treated with hydrochloric acid, bornylchloride, $C_{10}H_{16}HCl$, is formed. The latter is a camphor-like mass, fusing at 314.6° F.; it is readily decomposed, and under circumstances yields borneol during decomposition. Camphene has a peculiar turpentine-like and camphor-like odor, and exists in three modifications differing optically, namely: *terecamphene* or *levocamphene*, *austracamphene* or *dextrocamphene*, and *inactive camphene*.

*Limonene*, $C_{10}H_{16}$, forms the chief constituent of the volatile oils of the *Aurantiaceæ* (oils of lemon, orange peel, bergamot, etc.). It also occurs in oils of caraway, dill, nutmeg, pine needles, fleabane, etc. It is identical with *citrene*, *bergamene*, *hesperidene*, *carvene*, etc. It is a liquid terpene, with a lemon-like odor and specific gravity = 0.846 at 68° F. It boils at 347° to 348.8° F., and forms with great facility a solid tetrabromide, $C_{10}H_{16}Br_4$, which fuses at 219.2° to 221° F. and crystallizes in rhombic hemihedral crystals. It also yields a solid dihydrochloride, $C_{10}H_{16}2HCl$, which fuses at 120.2° to 122° F.; further, a dihydrobromide fusing at 147.2° F., and a dihydroiodide fusing at 172.4° F.; however, these three derivatives belong to the dipentene. By concentrated sulphuric acid limonene is almost completely converted into resin. To limonene belongs also a nitrosochloride, $C_{10}H_{16}NOCl$, which fuses at 217.4° F., and by boiling with alcoholic lye is converted into carvoscime fusing at 161.6° F. Carvol, $C_{10}H_{14}O$, may also be obtained from limonene nitrosochloride. Limonene yields a liquid monohydrochloride, $C_{10}H_{16}HCl$, which has a specific gravity of 0.98 and boils in vacuo at 194° F. Limonene exists in two modifications differing optically, namely, levo-limonene and dextro-limonene. The tetrabromides from both modifications are also optically isomeric. Mixtures of equal parts of dextro-limonene and levo-limonene yield dipentene.

*Dipentene*, $C_{10}C_{16}$, is contained in camphor oil and it appears also in cascarilla oil. It occurs also in all volatile oils which have been subjected to stronger heating and, moreover, is a product of the conversion of other terpenes; it may also be prepared from cineol, cajeput oil, caoutchouc, terpin, terpineol, etc. It is identical with cajeputene, cinene, isoterebenthene, caoutchene, diisoprene, terpilene, and perhaps also with divalerylene. It is a liquid of an agreeable lemon-like odor and boils at 356° to 357.8° F. Specific gravity $= 0.8538$ at 60.8° F. It yields a tetrabromide, $C_{10}H_{16}Br_4$, fusing at 257° F. It is decomposed by concentrated sulphuric acid, cymol $C_{10}H_{14}$ being formed. The dihydrochloride $C_{10}H_{16}2HCl$ fuses at 122° F., is insoluble in water, soluble in alcohol, and is decomposed by boiling alcohol. The dihydrobromide $C_{10}H_{16}2HBr$ fuses at 147.2° F., and the dihydriodide $C_{10}H_{16}2HI$ at 172.4° F. The monohydrochloride $C_{10}H_{16}HCl$ is obtained with difficulty; it boils at 194° to 197.6° F. and combines with $N_2O_4$. Dipentene exists in two modifications, both of which, as well as their derivatives, are inactive. By heating with alcoholic acid terpene is formed from dipentene.

*Sylvestrene*, $C_{10}H_{16}$. In Swedish and Russian oils of turpentine. It also occurs in the oils of *Pinus pumilio* and *Pinus sylvestris*, its presence in these oils having been proved beyond doubt by the formation of the typical hydrochloride with a fusing point of 161.6° F. Sylvestrene is a liquid terpene, possessing an odor of bergamot oil and boiling at 347° F. to 352.4° F. Specific gravity = 0.865 at 68.8° F. It yields a dihydrochloride $C_{10}H_{16}2HCl$, which fuses at 161.6° F. and in ethereal solution is dextrorotatory. The dihydrobromide also fuses at 161.6° F. and resembles the preceding combination; the dihydriodide, however, fuses at 150.8° to 152.6° F. The tetrabromide $C_{10}H_{16}Br$ forms monoclinic tables of 275° to 276.8° F. fusing point. Finally, there is a nitrosochloride $C_{10}H_{16}NOCl$ fusing at 222.8° to 224.6° F. Sylvestrene and its combinations are dextrorotatory.

*Terpinolene*, $C_{10}H_{16}$, is only a product of decomposition, and its presence in volatile oils has thus far not been established. It is formed by treating terpin, terpineol or cineol with sulphuric or phosphoric acid. It is a solid terpene fusing at 233.6° to 240.8° F. and boiling at 365° to 374° F. It yields a solid tetrabromide fusing at 233.6° to 235.4° F. By HCl or HBr dipentene derivatives are formed.

*Terpinene*, $C_{10}H_{16}$, occurs in cardamoms oil. It is formed by the action of sulphuric acid upon terpin, by boiling pinene with alcoholic sulphuric acid, etc. It is a liquid terpene of a lemon-like odor, boils at 354.2° to 359.6° F., and has a specific gravity = 0.855. Although it readily resinifies it is very constant. It forms a solid nitrite, $C_{10}H_{16}N_2O_3$, which fuses at 311° F.

*Phellandrene*, $C_{10}H_{16}$, occurs in fennel oil and water hemlock oil. It is a liquid terpene boiling at about 338° F. and yields a nitrite, $C_{10}H_{16}N_2O_3$, fusing at 201.2° F. It is very inconstant, and is converted by glacial acetic acid into dipentene, and by boiling with alcoholic sulphuric acid into terpinene. The phellandrene from the above-mentioned oils and from elemi oil is dextro-rotatory, and that from Australian eucalyptus oil levorotatory; the nitrites exhibit a rotatory power contrary to that of phellandrene.

*Sesquiterpenes*, $C_{15}H_{24}$, occur in the oils of cubebs, rosewood, patchouli, cloves, vetiver, sandal wood, cascarilla, calamus, cedar wood, galbanum, cade, savin, etc. The sesquiterpenes form a liquid boiling at 525.2° to 527° F.; they have a specific gravity of 0.921 at 68° F., and are levorotatory. They dissolve readily in ether, but with difficulty in alcohol and glacial acetic acid. They are decidedly inclined toward resinification. They yield a dihydrobromide, $C_{15}H_{24}2HBr$, which forms white needles fusing at 255.2° to 257° F.; also a dihydriodide, $C_{15}H_{24}2HI$, which crystallizes in needles and fuses at 221° to 222.8° F., being thereby decomposed; and further a solid dihydrochloride, $C_{15}H_{24}2HCl$, which fuses at 242.6° to 244.4° F. Solutions of sesquiterpenes (especially oxidized) in a large quantity of glacial acetic acid are colored green by a few drops of sulphuric acid, next indigo blue, and, when heated, red. There is a difference in the odor and boiling point of various sesquiterpenes; thus the sesquiterpene from Conina resin (from *Icica heptaphylla*) boils at 507.2° F., while the statements given above refer to the sesquiterpenes from the oils of cubebs, patchouli, cade, etc.

*Diterpenes*, $C_{20}H_{32}$, occur in copaiva, and may be obtained from oil of turpentine and colophony, as well as from caoutchouc. The diterpene obtained from Para copaiva is a liquid resembling in odor the balsam and boiling at 485.6° to 500° F. In the presence of moisture the diterpene, when rectified over sodium, yields a deep blue fluid of the formula $3C_{20}H_{32},H_2O$, which boils at 485.6° to 500° F. By allowing sulphuric acid or phosphoric oxide to act upon oil of turpentine, or subjecting colophony to destructive distillation, *colophene*—an oily, sticky diterpene boiling at 604.4° to 608° F.—passes over, while caoutchouc or gutta-percha yields, by destructive distillation, a diterpene—heveene—which is a liquid of slight odor and pungent taste, and boils at 599° F.

*Polyterpenes*, $(C_{10}H_{16})_2 + x$, have thus far not been closely examined. They occur separately in volatile oils and form the chief constituent of caoutchouc. By destructive distillation caoutchouc yields isoprene, $C_5H_8$, dipentene, $C_{10}H_{16}$, and heveene, $C_{20}H_{32}$.

Next to the hydrocarbons, *alcohols* and *phenols* have to be mentioned as constituents of volatile oils. Alcohols as well as phenols are hydroxides.

Of the alcohols have to be specified:

*Methyl alcohol*, $CH_3.OH$, commonly called *wood spirit*. It occurs in small quantities in heracleum (cow parsnip) oil. The wintergreen oil from *Gaultheria procumbens* consists chiefly of the methyl ether of salicylic acid. On distilling this oil with caustic potash, pure methyl alcohol is obtained. Methyl alcohol is a colorless, mobile fluid, possessing a pure spirituous smell. Its specific gravity is 0.8142 at 32° F. It boils at 149.2° F. It is miscible in all proportions with water. When ignited it burns with a pale blue flame.

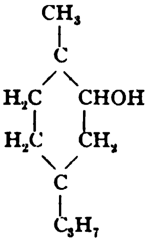
*Octyl alcohol* (*normal*), $C_8H_{17}OH$, occurs in small quantities in the volatile oil of the seeds of *Heracleum spondylium*, the cow-parsnip. It is a colorless oily liquid of a penetrating aromatic odor, boils at 384.8° to 386.6° F., and has a specific gravity of 0.83 at 68° F.

*Benzyl alcohol*, $C_6H_5CH_2OH(=C_7H_8O)$, occurs in small quantities in Peru balsam and perhaps also in cherry laurel oil. It is a liquid boiling at 402.8° F. and possesses a slightly aromatic odor. Specific gravity $= 1.063$ at 32° F. It is soluble up to 4 per cent. in water at 62.6° F.

*Borneol*, $C_{10}H_{17}OH(=C_{10}H_{18}O)$, also called *bornyl alcohol*, *camphyl alcohol* or *Borneo camphor*. This camphor is found in the old trunks of *Dryobalanops camphora*, *D. longifolia* and *D. becarii*. It occurs in borneo camphor oil and the following volatile oils: Snake root, feverfew, lavender, spike and rosemary. Two modifications of it are known, namely, dextro-borneol (ordinary Borneo camphor) and levo-borneol. The latter is derived from *Blumea balsamifera*, D. C., indigenous to Haïnan, and is called *Ngai camphor*. It also occurs in the oils of feverfew, lavender, spike and rosemary.

Borneol forms camphor-like pieces smelling like camphor, though also slightly of pepper or patchouli. It is more friable than ordinary camphor, very sparingly soluble in water, but readily so in alcohol and ether. From petroleum-ether it crystallizes in excellent table-like crystals. Fusing point 402.8° to 404.6° F.; boiling point, 413.6° F. It is, however, volatile below the fusing point, subliming thereby in beautiful hexagonal laminæ. Dextro-borneol and levo-borneol differ only in the direction of their rotation. Borneol may be prepared *artificially* from camphor. The best method for this purpose is the one given by O. Wallach: In a large flask dissolve 50 grammes of camphor ($C_{10}H_{16}O$) in 500 cubic centimeters of 96 per cent. alcohol, and place upon the flask a reflux-condenser with a wide condensing tube. Through the condensing tube introduce gradually into the flask 60 grammes of sodium in small pieces, about an hour being required for this operation. The fluid becomes warm at first more rapidly and then more slowly. When warming abates add, with vigorous shaking, about 50 cubic centimeters of water to accelerate the solution of the sodium. When all the sodium is consumed, pour the contents of the flask into 3 or 4 quarts of cold water, allow to settle, and collect the separated borneol upon a straining cloth, upon which it is freed as much as possible from adhering alkali by washing with water. Next dry the borneol by spreading it out upon unglazed plates, and finally recrystallize it from petroleum ether. The borneol separates in excellent, mostly table-like crystals.

The constitution of borneol is generally expressed by the following formula, which is that of a secondary alcohol:



*Terpin hydrate*, $C_{10}H_{18}(OH_2)+H_2O$, also called *camphor of oil of turpentine* or *dipentenyl glycol*. It occurs in the oils of sweet basil and cardamoms, as well as in old oil of turpentine. It forms large, transparent, monoclinic prisms soluble in 200 parts of cold or 22 parts of boiling water, but more readily so in alcohol. It fuses at 240.8° to 242.6° F. By distillation it loses first its water of crystallization, after which the anhydrous glycol, called terpin, commences to boil at 496.4° F. and distills off. The distillate solidifies to a hard mass, fusing at 215.6° F., which is very hygroscopic and readily combines with water. According to G. Vulpius, 1 part of terpin hydrate dissolves, at 59° F., in 200 parts of water, 100 parts ether, and 200 parts chloroform, 10 parts alcohol, or in 32 parts boiling water and 2 parts boiling 90 per cent. alcohol. Finely-powdered terpin hydrate spread out and exposed to the air becomes odorless, and remains so when re-crystallized from hot alcohol.

*Terpineol*, $C_{10}H_{17}OH$, also called *terpinol* and *dipentenyl alcohol*, occurs in oil of cardamoms. It is a thick fluid of an agreeable odor, boiling at 419° F. and solidifying at —58° F. Terpineol is a non-saturated alcohol isomeric with borneol, but while the latter may have originated from camphene, the former belongs to dipentene.

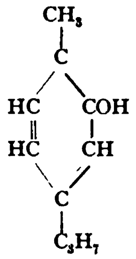
*Menthol*, $C_{10}H_{19}OH$, also called *menthyl alcohol* and *peppermint camphor*, forms the most important constituent of peppermint oil. It crystallizes in prisms, has a strong odor of peppermint, fuses at 107.6° F. and boils at 413.6° F. It is levo-rotatory. Menthol seems to be a secondary alcohol.

*Tanacetyl alcohol*, $C_{10}H_{17}OH$ occurs in tansy oil. It is a primary alcohol, non-saturated (a double combination). It is a liquid boiling at 401° F., the aldehyde of which seems to be the tanacetol $C_{10}H_{16}O$. Tanacetyl alcohol is isomeric with borneol.

Of the *phenols* as constituents of volatile oils, the following have to be mentioned:

*Thymol*, $C_6H_3OH(CH_3.C_3H7) = C_{10}H_{13}OH$, also called *metacymophenol*, forms the chief constituent of thyme oil, from which it is prepared. It crystallizes in large, monometrical or asymmetrical crystals of a thyme odor and sharp pungent taste. Fusing point 122° F.; boiling point 428° to 446° F.

*Carvacrol*, $C_6H_3(OH)C_3H_7CH_3 = C_{10}H_{14}O$ (*orthocymophenol*), occurs in the oils of origanum, summer-savory, caraway, wild thyme, etc. It is a thickish liquid of an aromatic odor; it solidifies in the cold, boils at 458.6° F., is isomeric with carvol, but very likely of the following constitution:

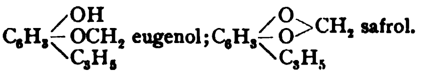


In connection with the alcohols and phenols the *ethers* may be discussed.

*Anethol or allyl-phenol-methyl ether,* $C_6H_4(OCH_3)C_3H_5=C_{10}H_{12}O$, occurs in the oils of fennel, aniseed, pimpinella (saxifrage), star anise, etc.; it crystallizes from them on cooling. It smells of anise. Fusing point 69.8° F.; boiling point 449.6° F.; specific gravity = 0.989 at 82.4° F. It is inclined towards polymerism.

*Eugenol or eugenic acid,* $C_6H_3OH(OCH_3)C_3H_5=C_{10}H_{12}O_2$, occurs in the oils of cloves, cinnamon leaves, cinnamon root, etc. It is a very refractive liquid, possessing the odor and taste of cloves. When exposed to the air it turns brown. Boiling point 477° F.; specific gravity = 1.063 at 64.4° F. It is highly antiseptic. *Methyleugenol,* $C_6H_3(OCH_3)_2C_3H_5=C_{11}H_{14}O_2$, occurs in wild ginger oil. It is a liquid boiling at 471.2° to 473° F.

*Safrol,* $C_{10}H_{10}O_2$, is not an ether, but stands in a certain relation to eugenol, as shown by the following formulæ:



$$C_6H_3\begin{cases}OH\\OCH_2\\C_3H_5\end{cases}\text{eugenol};\ C_6H_3\begin{cases}O\\O\\C_3H_5\end{cases}\!\!\!\!\!\!\!\!\!\!\!\!\!>CH_2\ \text{safrol.}$$

Occurs in the oils of sassafras, shikimi, etc. Refractive monoclinic crystals which fuse at 45.4° F. to a liquid possessing an agreeable, aromatic odor and boiling at 449.6° F. It solidifies again at — 13° F.

*Asaron*, $C_6H_2(OCH_3)_3C_3H_5=C_{12}H_{16}O_3$, occurs in wild ginger oil. Odorless monoclinic crystals of a slightly pungent taste. Fusing point, 138.2° F.; boiling point, 564.8° F. Sparingly soluble in hot water, but readily soluble in alcohol and ether.

In connection with ethers may be mentioned:

*Cineol*, $C_{10}H_{18}O$, isomeric with borneol. It forms the principal constituent of the oils of wormseed, cajeput and eucalyptus, and by reason of its occurrence in the last mentioned oils is also called *cajeputol* or *eucalyptol*. It is a liquid of an agreeable, camphor-like odor, inactive, boils at 348.8° to 350.6° F. Specific gravity = 0.923 at 60.8° F. It solidifies in the cold, and then fuses again at 30.2° F. By warm sulphuric acid cineol is converted into terpinene and terpinolene. It yields a dibromide $C_{10}H_{18}OBr_2$, which crystallizes in red crystals; further a hydrobromide $C_{10}H_{18}OHBr$, a white crystalline precipitate, which fuses at 132.8° to 134.6° F.

*Products of oxidation appearing in volatile oils.* These are aldehydes and acids, as well as ketones. Of these three groups of combinations a larger number has to be mentioned as constituents of volatile oil. First, *the aldehydes:*

*Benzaldehyde*, $C_6H_5COH = C_7H_6O$, occurs in the oils of bitter almond, peach kernel, cherry laurel, etc. It is a colorless, very refractive liquid, possessing an odor of bitter almonds and a pungent aromatic taste. It is soluble in 300 parts of water. Specific gravity = 1.0636 at 32° F. and 1.0504 at 59° F. Boiling point 354.2° F. Oxidizes readily in the air to benzoic acid, $C_6H_5COOH$.

*Salicylaldehyde* $C_6H_4OHCOH = C_7H_6O_2$ exists in the oils of meadow sweet and of other species of *Spiraea*. It is a liquid possessing an agreeable fragrant smell and a pungent aromatic taste. Specific gravity = 1.1731 at 56.3° F. Boiling point 385.9° F. Solidifies at —4° F.

*Cuminaldehyde* or *cuminol*, $C_6H_4C_3H_7COH = C_{10}H_{12}O$, exists in the oils of Roman cumin, water hemlock, etc. It is an oily liquid, having an odor of Roman cumin oil. It oxidizes readily. Boiling point, 457.9° F.

*Cinnamaldehyde*, $C_6H_5C_2H_2COH$, exists in the oils of cinnamon and cassia. It is a colorless liquid of a very fragrant cinnamon-like odor. It volatilizes without decomposition only in *vacuo*, or with steam. It boils at 266° F. It absorbs readily oxygen, and is converted into cinnamic acid. Dilute nitric and chromic acids oxidize it to benzaldehyde and benzoic acid.

The number of *acids* occurring in volatile oils is considerably greater:

*Isobutyric acid*, $C_3H_7COOH = C_4H_8O_2$, occurs in arnica flower oil and as ester in the oils of Roman chamomile and arnica. It is a liquid possessing a rancid odor, and boiling at 309.2° F. Specific gravity = 0.9598 at 32° F. Soluble in 3 parts of cold water.

*Valerianic acid*, $C_4H_9COOH = C_5H_{10}O_2$, occurs in the oils of valerian and angelica; also frequently as ester. It is an inactive, mobile oily liquid of specific gravity 0.9536 at 32° F., and boils at 347° F. It has a sour smell and a pungent cheese-like odor. Soluble in 30 parts of water.

*Oxymyristic acid*, $C_{13}H$ OHCOOH = $C_{14}H_{28}O_3$ (derived from myristic acid $C_{13}H_{27}COOH$) in angelica oil. It forms laminæ of a nacreous lustre, which fuse at 123.8° F.

*Angelic acid*, $C_4H_7COOH = C_5H_8O_2$, in sumbul root oil; frequently occurs also as ester. It forms long monoclinic prisms of an aromatic odor. Fusing point 111.2° to 113° F.; boiling point 365° F. Soluble with difficulty in cold, but readily so in hot, water.

*Benzoic acid*, $C_6H_5COOH = C_7H_6O_2$, in gum benzoin, tolu balsam, Peru balsam, myrrh, storax; free, or as ester, in ylang-ylang oil. Benzoic acid crystallizes from a hot aqueous solution in large, thin, flexible, and brilliant needles or plates, fusing at 248° F. It boils at 482° F. but sublimes readily at a much lower temperature (about 284° F.), and volatilizes by boiling its solution. The vapor has an aromatic odor, is irritating, and produces coughing, and attacks the eyes. Benzoic acid is only sparingly soluble in cold water, but readily in boiling water and alcohol. Its properties are considerably changed by very small quantities of impurities.

*Salicylic acid*, $C_6H_4OHCOOH = C_7H_6O_3$, also called *oxybenzoic acid*, occurs in small quantities in the oil of several species of *Spiræa*, and in the form of ester in the oil of wintergreen. It crystallizes in fine needles or monoclinic prisms of a slightly astringent, sweetish sour taste. It is sparingly soluble in water, but very readily in alcohol and ether. Fusing point 311° to 312.8° F. It can be sublimed, and forms an excellent antiseptic.

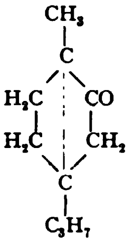
*Cinnamic acid or phenylacrylic acid*, $C_6H_5C_2H_2COOH = C_9H_8O_2$, in liquid storax, in balsams of Peru and tolu, and frequently in the form of esters. It crystallizes from boiling water in fine needles fusing at 271.4° F., and boiling at 572° to 579.2° F. It is scarcely soluble in cold, and with great difficulty in hot, water.

Finally a few *ketones* have to be mentioned:

*Methyl-monyl ketone*, $CH_3COC_9H_{19} = C_{11}H_{22}O$, forms the chief constituent of oil of rue. It is a colorless, very refractive, oily liquid possessing an odor of rue oil and boiling at 435.2° F. Specific gravity = 0.8295 at 63.5° F. In the cold it solidifies to a crystalline mass which fuses again at 59° to 60.8° F.

*Camphor*, $C_{10}H_{16}O$, also called *common*, *Japan*, or *Chinese camphor*, in camphor oil, in the oils of spike, lavender, rosemary and sage. Pure camphor fuses at 347° F. and boils at 399.2° F., but is very volatile at the ordinary temperature. At 68° F. 1 part camphor dissolves in 1300 parts water, and at 53.6° F., in 0.8 part alcohol. Specific gravity = 0.806. It is readily soluble in ether, chloroform, benzol, glacial acetic acid, and carbon disulphide. Common camphor in its solutions is dextro-rotatory. However, there is also a levo-camphor obtained by oxidation of camphene, and an inactive camphor. Camphor can be pulverized only by previously moistening it with a solvent (alcohol). Small pieces of camphor thrown on water move about with a revolving motion.

While borneol has been obtained from camphor by a reducing action, camphor is formed from borneol by oxidation. Now, since borneol is a secondary alcohol, the product of oxidation—camphor—must be a ketone. It has very likely the following formula:



*Carvol*, $C_{10}H_{14}O$, occurs in the oils of caraway, fennel and dill. It is a liquid, possessing an odor of caraway, and boiling at 435.2° to 437° F. It is dextro-rotatory, only the carvol from peppermint oil being levo-rotatory. It is readily converted into the isomeric carvacrol (= orthocymophenol), and into a ketone derived from limonene.

Closely connected with the products of oxidation of the alcohols (aldehydes, acids, ketones) are a few derivatives of acids. They are chiefly *esters* or *compound ethers* and *nitriles*. Among the esters have to be mentioned:

*Esters of butyric and isobutyric acids*. The former occur, for instance, in the volatile oils of a few *Umbelliferæ* and *Compositæ*, and the latter in the oils of Roman chamomile and arnica flowers.

*Ester of caproic acid* in heracleum (cow-parsnip) oil.

*Ester of angelic acid* in the oils of angelica, sumbul root, Roman chamomile, etc.

*Methyl-ester of salicylic acid*, $C_6H_4OHCOOCH_3 = C_8H_8O_3$ in the oils of wintergreen and birch. It is obtained artificially by distilling salicylic acid with methyl alcohol and sulphuric acid. It forms a liquid possessing an agreeable odor and a refreshing aromatic taste. Boiling point 422.6° F.

*Phlorol-ester of isobutyric acid*, $C_3H_7COOC_8H_9 = C_{12}H_{16}O_2$, a liquid occurring in arnica root oil and boiling at 435.2° to 437° F. The radicle $C_8H_9$ is ethylized phenyl ($C_6H_4C_2H_5$).

*Styracin*, $C_6H_5C_2H_2COO(C_6H_5C_3H_4)$ = *cinnyl cinnamate*, occurs in liquid storax. By crystallization from alcohol it is obtained in white tufts or prisms which fuse at 111.2° F.

Of *nitriles* the following occur in volatile oils:

*Benzonitrile,* $C_6H_5CN$, in Indian or garden cress oil. It is a colorless, oily, and refractive liquid, boiling at 375.8° F., and smelling like bitter almonds. Specific gravity = 1.023 at 32° F. On exposure to a very low temperature it solidifies, and then fuses again at 1.4° F. It is soluble in 100 parts water, but more readily in alcohol and ether. It is derived from benzoic acid, $C_6H_5COOH$.

*Phenylacetonitrile,* $C_6H_5CH_2CN = C_8H_7N$, a water-clear liquid of a peculiar odor, and boiling at 449.6° F. It occurs in oil of cress. It is derived from phenylacetic acid, $C_6H_5CH_2COOH$.

*Phenylpropionnitrile,* $C_6H_5C_2H_4CN = C_9H_9N$ ($\beta$ combination) forms the chief constiteunt of water cress oil. It is a liquid boiling at 501.8° F. It is derived from $\beta$-phenylpropionic or hydrocinnamic acid, $C_6H_5C_2H_4COOH$.

*Phenyloxyacetonitrile,* $C_6H_5CHOHCN = C_8H_7ON$, also called *nitrile of mendelic acid,* occurs in bitter almond oil containing prussic acid. It is an oily liquid which solidifies at 14° F., and at 338° F. decomposes to prussic acid and benzaldehyde. It is insoluble in water but readily soluble in alcohol and ether. It is derived from mandelic acid, $C_6H_5CHOHCOOH$.

It remains to mention the *sulphuretted constituents of volatile oils.* They are partially *sulpho-ethers* or *thio-ethers, i. e.,* sulphides of alcohol radicles, partially *thiocarbimides* or *isosulphocyanates,* hence esters.

The group $(CN)_1$ is either nitrile—$C \equiv N$ or carbamine $C = N$. Combinations of the latter are also called carbamine or isocyanic combinations. Analogically to this the acid $N \equiv C—(OH)$ is called *cyanic acid,* and the acid $C = N—(OH)$ *isocyanic acid.* The salts of the latter are also called carbimides. Now $N \equiv C—(SH)$ is *sulphocyanic* or *thiocyanic acid,* and, on the other hand, $C = N—(SH)$ *isosulphocyanic acid,* the salts and esters of which are also called *thiocarbimides.* Ordinarily they are called *mustard oils.*

*Allylsulphide*, $(C_3H_5)_2S = C_6H_{10}S$, occurs in the oils of garlic and onion. It is a colorless liquid of high refractive power, boiling at 284° F. and possessing the peculiar odor of garlic in the highest degree.

*Vinyl sulphide*, $(C_2H_3)_2S = C_4H_6S$, occurs in oil of *Allium ursinum*.

*Butylthiocarbimide*, $C_4H_9CNS = C_5H_9NS$, in the volatile oil of the scurvy grass (*Cochlearia officinalis*). It is a liquid having a very pungent taste and smell, and boiling at 320° F.

*Allylthiocarbimide*, $C_3H_5CNS = C_4H_5NS$, in mustard oil. It is a colorless, highly refractive liquid. Specific gravity = 1.036 at 32° F. Boiling point 303° F. Taste and odor penetrating, irritating, producing tears.

In conclusion a few *glucosides*, by which are understood a class of bodies very widely diffused in the vegetable kingdom, which are resolved by the action of ferments into glucose, and some other substance. They do not occur in volatile oils, but in the plants from which volatile oil is obtained by their decomposition by ferments. The *potassium salt of myronic acid*, $C_{10}H_{18}KNS_2O_{10}$ exists in the seed of the black mustard and crystallizes in silky needles, but may also be obtained in short glassy prisms. It is odorless, has a cooling bitter taste, and is soluble in water and in alcohol. By the action of a ferment called *myrosin*, which is contained in the seed, it splits up into mustard oil, glucose, and acid potassium sulphate.

The glucoside *sinalbin*, $C_{30}H_{44}N_2S_2O_{16}$, occurs in white mustard seed, and forms a substance crystallizing in small glassy needles readily soluble in water, but with difficulty in alcohol. By myrosin it also suffers a peculiar decomposition, the principal products of which are mustard-seed oil and glucose.

*Amygdalin*, $C_{20}H_{27}NO_{11} + 3H_2O$, occurs in the kernels of cherries, plums, apricots, etc., and in large quantities in bitter almonds and the leaves of the common laurel; it exists also in the leaves of the bird-cherry and the sprouts and bark of the mountain-ash, and in other plants belonging to the family of *Drupaceæ* and *Amygdaleæ*. It crystallizes in pearly laminæ and scales, which lose their water at 248° F., fuse at 392° F., and are soluble in 12 parts of water at 47° to 54° F., and very readily so in boiling water. When ***emulsin*** (the ferment of bitter almonds) is added to the aqueous solution of amygdalin, it splits up into prussic acid, benzaldehyde and glucose. Amygdalin at first tastes sweet, then bitter, and is poisonous. Some constituents of volatile oils are not mentioned here, it being impossible to classify them in one or the other group. They constitute especially a few of the so-called camphor varieties, of which may be mentioned:

$C_{10}H_{16}O$ (*isomeric with common camphor*): ***alantol*** in elecampane oil; ***salviol*** in sage oil; ***myristicol*** in nutmeg oil; ***pulegiol*** in pennyroyal oil, a liquid having a peppermint-like odor and boiling at 440.6° F.; ***thujol*** in thuja oil.

$C_{10}H_{18}O$ (*isomeric with borneol*): ***geraniol*** in geranium oil; ***coriandrol*** in coriander oil.

*Matico camphor*, $C_{12}H_{20}O$, in matico oil; ***gurjunol***, $C_{20}H_{28}(OH)_2$, in gurjun oil.

$C_{15}H_{26}O$ (*sesquihydroterpene*): ***cubebs-camphor*** in cubebs oil; ***cedar-camphor*** in cedar oil; ***ledum-camphor*** in wild rosemary or marsh tea oil; ***patchouly-camphor*** in patchouli oil; ***santatol*** in sandal-wood oil.

Full information regarding the constituents of a large number of volatile oils is found in the appended table. The first column to the left contains the Latin names of the plants from which the oils mentioned in the second column are derived, as well as the names of the natural families of plants to which the respective plants belong. The third and fourth columns contain the limits of value for the specific gravities and boiling points. Finally, the last column contains statements regarding the chemical composition of the respective volatile oils, the components in italics constituting the chief mass.

Derivation, Specific Gravity, Boiling Point, and Chemical Constitution of Volatile Oils.

| Family, Variety. | Name of the Oil. | Specific gravity. | Boiling point, ° F. | Chemical Constitution. |
|---|---|---|---|---|
| *Coniferæ:* | | | | |
| Thuja occidentalis, L. | Thuja oil | 0.91–0.93 | 320–482 | *Thujol*, $C_{10}H_{16}O$ (90 per cent.); terpene, $C_{10}H_{16}$ (10 per cent). |
| Juniperus communis, L. | Juniperberry oil | 0.87–0.88 | 339.8–359.6 | Juniper camphor, $C_{10}H_{16}O,H_2O$; levo-pinene, $C_{10}H_{16}$ (33 per cent). |
| Juniperus communis, L. | Juniperwood oil | 0.87 | — | |
| Juniperus Oxy-cedrus, L. | Cade oil | — | — | Sesquiterpene, $C_{15}H_{24}$. |
| Juniperus Sabina, L. | Savin oil | 0.89–0.91 | 311–321.8 | Terpene, $C_{10}H_{16}$; sesquiterpene, $C_{15}H_{24}$. |
| Juniperus Virginiana, L. | Cedarwood oil | 0.95–0.96 | 519.8–527 | *Cedrene*, $C_{15}H_{24}$; cedar camphor, $C_{15}H_{26}O$. |
| Sequoia gigantea, Torr | Sequoia oil | — | — | *Terpene*, $C_{10}H_{16}$; sequojene, $C_{13}H_{10}$. |
| Pinus pinaster, Sol | French oil of turpentine. | 0.864 | 312.8–321.8 | *Levo-pinene*, $C_{10}H_{16}$; dipentene, $C_{10}H_{16}$. |
| Pinus australis, Michx | American oil of turpentine | 0.864 | 312.8–321.8 | *Dextro-pinene*, $C_{10}H_{16}$; dipentene, $C_{10}H_{16}$. |
| Pinus sylvestris, L., Ledebourii, End | Russian oil of turpentine. | 0.86–0.87 | 334.4–356 | Dextro-pinene and sylvestrene, $C_{10}H_{16}$; dipentene. |
| Pinus abies, L. | Red pine oil | — | 332.6 | Terpenes, $C_{10}H_{16}$. |
| Pinus sibirica | Siberian oil of turpentine. | — | 332.6 | Terpene, $C_{10}H_{16}$. |
| Pinus Sabiniana, Dougl | Nut pine oil | 0.694 | 213.8 | *Heptane*, $C_7H_{16}$. |
| Pinus Picea, L | Spruce fir oil | 0.856 | 341.6 | *Levo-pinene*, $C_{10}H_{16}$. |
| Pinus sylvestris, L | Pine leaf oil | 0.89 | 318.2–356 | *Levo-pinene*, $C_{10}H_{16}$; levo-limone; dipentenes. |
| Pinus Pumilio, Haenke | Dwarf pine oil | 0.983 | 312.8–482 | Levo-pinene; sesquiterpene, $C_{15}H_{24}$; sylvestrene. |
| *Liliaceæ:* | | | | |
| Allium sativum, L. | Garlic oil | 1.02–1.05 | 302 | *Allyl-sulphide* $(C_3H_5)_2S$; allyl-oxide $(C_3H_5)_2O$. |
| Allium Cepa, L. | Onion oil | 1.036 | — | *Allyl-sulphide* $(C_3H_5)_2S$; allyl-oxide $(C_3H_5)_2O$. |

DERIVATION, SPECIFIC GRAVITY, BOILING POINT, AND CHEMICAL CONSTITUTION OF VOLATILE OILS.—*Continued.*

| Family, Variety. | Name of the Oil. | Specific gravity. | Boiling point, ° F. | Chemical Constitution. |
|---|---|---|---|---|
| Allium ursinum, L. | Long-rooted garlic oil | 1.015 | 204.8–284 | *Vinylsulphide* $(C_2H_3)_2S$ (67 per cent.); vinylpolylsulphide. |
| *Amaryllidaceæ:* | | | | |
| Narcissus Jonquilla | Jonquil oil | .... | 212 | Jonquil camphor. |
| *Iridaceæ:* | | | | |
| Iris florentina, L. | Orris root oil | — | — | *Myristic acid*, $C_{14}H_{28}O_2$. |
| Crocus sativus, L. | Saffron oil | .... | 406.4–410 | $C_{20}H_{14}O_2$? |
| *Araceæ:* | | | | |
| Acorus Calamus, L. | Calamus oil | 0.93–0.99 | 410–554 | *Pinene*, $C_{10}H_{16}$; terpene. |
| *Gramineæ:* | | | | |
| Andropogon Nardus, L. | Citronella oil | 0.89–0.897 | 415.4–426.2 | *Citronellol*, $C_{10}H_{18}O$; terpene; alcohol, $C_{10}H_{20}O$. |
| Andropogon Schoenanthus, L. | Lemon grass oil | 0.87–0.898 | 392–431.6 | *Geraniol*, $C_{10}H_{18}O$; terpene, $C_{10}H_{10}$; valerianic acid, $C_5H_{10}O_2$. |
| Andropogon pachnodes | Indian geranium oil | 0.88–0.89 | 410–417.2 | |
| Andropogon muricatus, Retz. | Vetiver oil | 1.007 | — | — |
| *Zingiberaceæ:* | | | | |
| Zingiber officinalis, Rosc. | Ginger oil | 0.893 | 492.8 | *Sesquiterpene*, $C_{15}H_{24}$; terpene; cymol. |
| Alpinia officinarum, Hance | Galangal oil | 0.85–0.92 | 338–527 | Cineol, $C_{10}H_{18}O$. |
| Curcuma longa, L. | Turmeric oil | $<1$ | 428–482 | *Curcumol*, $C_{10}H_{14}O$; Phellandrene, $C_{10}H_{16}$. |
| Curcuma Zedoariæ, Rosc | Zedoary oil | $>1$ | | |
| Elettaria Cardamomum, Wh. | Cardamoms oil | 0.92–0.95 | 338–428 | *Terpineol*, $C_{10}H_{18}O$; terpenes, $C_{10}H_{16}$; dipentene. |
| Amomum Melegueta, Rosc. | Grains of Paradise oil | 0.825 | 455–496.4 | $C_{20}H_{32}O$? |

DERIVATION, SPECIFIC GRAVITY, BOILING POINT, AND CHEMICAL CONSTITUTION OF VOLATILE OILS.—*Continued.*

| Family, Variety. | Name of the Oil. | Specific gravity. | Boiling point, ° F. | Chemical Constitution. |
|---|---|---|---|---|
| *Cupuliferæ:* | | | | |
| Betula alba, L. | Birch tar oil | 0.847 | 312.8 | *Terpene*, $C_{10}H_{16}$. |
| Betula lenta | Birch bark oil | 1.03–1.18 | 422.6–444.2 | *Methyl salicylate*, $C_8H_8O_3$ (100 per cent). |
| Fagus sylvatica, L. | Beech tar oil | 0.98 | 302–482 | Phenoles (33 per cent). |
| *Myriaceæ:* | | | | |
| Myrica Gale, L. | Sweet gale oil | 0.876 | — | *Stearoptene*, (70 per cent). |
| *Saliaceæ:* | | | | |
| Populus nigra, L | Poplar buds oil | 0.90 | 491–509 | *Diterpene*, $C_{16}H_{24}$. |
| *Urticaceæ:* | | | | |
| Cannabis sativa, L. | Hemp oil | 0.93 | 492.8–496.4 | *Sesquiterpene*, $C_{15}H_{24}$. |
| Humulus lupulus, L. | Hop oil | 0.908 | 257–455 | *Camphor*, $C_{10}H_{18}O$ (67 per cent.); terpene, $C_{10}H_{16}$ (17 per cent). |
| *Piperaceæ:* | | | | |
| Piper nigrum, L. | Pepper oil. | 0.86–0.99 | 332.6–356 | Phellandrene; terpene, $C_{10}H_{16}$. |
| Piper longum, L. | Long pepper oil | 0.861 | 482–572 | |
| Piper Bette, L. | Betel oil from fresh leaves. | 0.969 | 343.4–509 | *Sesquiterpene*, $C_{15}H_{24}$; *terpene*, $C_{10}H_{16}$; chavicol, $C_9H_{10}O$; betel-phenol. |
| Piper Bette, L. | Betel oil from dry leaves. | 1.024 | 392–527 | *Methoxylchavicol*, $C_{10}H_{12}O_2$ (70–75 per cent.); sesquiterpene. |
| Piper angustifolium, R | Matico oil | 0.953 | 392 | Matico camphor, $C_{12}H_{20}O$ (little). |
| Piper Cubeba, L. | Cubebs oil | 0.92–0.93 | 482–500 | *Sesquiterpene;* terpene; cubebs camphor $C_{15}H_{26}O$. |
| *Chenopodiaceæ:* | | | | |
| Chenopodium ambrosioides, L. | Mexican tea oil | 0.9 | 356 | |

DERIVATION, SPECIFIC GRAVITY, BOILING POINT, AND CHEMICAL CONSTITUTION OF VOLATILE OILS.—*Continued.*

| Family, Variety. | Name of the Oil. | Specific gravity. | Boiling point, ° F. | Chemical Constitution. |
|---|---|---|---|---|
| *Lauraceæ :* | | | | |
| Laurus nobilis, L. | Bayberry oil | 0.91–0.93 | 338–482 | *Cineol*, $C_{10}H_{18}O$; levo-pinene; sesquiterpene. |
| Laurus nobilis, L. | Bay-leaves oil | 0.924 | 316.4 | *Cineol*, $C_{10}H_{18}O$; levo-pinene, $C_{10}H_{16}$. |
| Ocotea varieties | Guayama laurel oil | 0.864 | — | *Pinene*, $C_{10}H_{16}$. |
| Nectandra Puchury, N. M | Pichurim bean oil | — | — | Oxygenous bodies. Fatty acids. |
| Laurus Sassafras, L. | Sassafras oil | 1.06–1.09 | 429.8–447.8 | *Safrol*, $C_{10}H_{10}O_2$ (90 per cent.); Safrene, $C_{10}H_{10}$ (10 per cent). |
| Cinnamomum aromaticum, Nees | Cassia oil | 1.05–1.07 | 485.6–491 | *Cinnamaldehyde*, $C_9H_8O$ (70 to 90 per cent.); cinnamic acid, $C_9H_8O_2$; cinnamacetate, $C_{11}H_{12}O_2$. |
| Cinnamomum zeylanicum, Breyn | Cinnamon oil | 1.02–1.06 | 428–464 | Same as preceding; besides eugenol, $C_{10}H_{12}O_2$ (4 to 8 per cent). |
| Cinnamomum zeylanicum, Breyn | Cinnamon leaves oil | 1.053 | .... | *Eugenol*, $C_{10}H_{12}O_2$ (90 per cent.); terpene, $C_{10}H_{16}$ (10 per cent). |
| ? | Cinnamon root oil | .... | .... | *Eugenol* (50–70 per cent); safrol, $C_{10}H_{10}O_2$; benzoic acid ester. |
| Cinnamomom Kiamis, Nees? | Massoy bark oil | 1.05 | 392–572 | *Eugenol* (80 per cent.); pinene; limonene; dipentene, $C_{10}H_{16}$. |
| Cinnamomum Culilawan, Nees | Culilawan oil | | | |
| Lindera sericea, Bl. | Kuro-moji oil | | | |
| Laurus Camphora, L. | Japan camphor oil | 0.9–0.92. or 0.96–0.97 | 347, or 464–572 | Terpenes; sesquiterpenes; camphor varieties. |
| Dryobalanops Camphora, C. | Borneo camphor oil | 0.95 | 356–572 | Terpene; sesquiterpene; camphor, $C_{14}H_{22}O$. |
| Oreodaphne Californica | California bay laurel oil. | 0.94 | 332.6–500 | Terpinol, $C_{20}H_{34}O$; umbellol, $C_8H_{12}O$. |
| Laurus Benzoin, L. | Spice wood oil | 0.855–0.923 | 320–392 | *Hydrocarbons ;* methyl salicylate, $C_8H_8O_3$. |
| ? | Parakoto bark oil | 0.93 | 172.4–482 | Parakotene, $C_{11}H_{18}$; parakotol, $C_{15}H_{24}O$ and $C_{28}H_{40}O_8$. |

DERIVATION, SPECIFIC GRAVITY, BOILING POINT, AND CHEMICAL CONSTITUTION OF VOLATILE OILS.—*Continued.*

| Family, Variety. | Name of the Oil. | Specific gravity. | Boiling point, ° F. | Chemical Constitution. |
|---|---|---|---|---|
| *Myristicaceæ:* | | | | |
| Myristica moschata, Thunbg. .... | Mace oil .............. | 0.89–0.95 | 347–392 | *Pinene*, $C_{10}H_{16}$; dipentene; myristicine, $C_{12}H_{14}O_3$ (22 per cent.); myristic acid, $C_{14}H_{28}O_2$. |
| Myristica moschata, Thunbg. .... | Nutmeg oil ............ | 0.88–0.95 | 339.8–341.6 | *Terpenes*, $C_{10}H_{16}$; cymol, $C_{10}H_{14}$; myristicol, $C_{10}H_{16}O$. |
| *Anonaceæ:* | | | | |
| Cananga odorata, H. et Th. ...... | Ylang-ylang oil ........ | 0.947–0.974 | 320 | Benzoic acid ester; phenol; aldehyde or ketone. |
| *Magnoliaceæ:* | | | | |
| Illicium anisatum, Lour. ........ | Star anise oil .......... | 0.976–0.978 | — | Anethol, $C_{10}H_{12}O$; terpene, $C_{10}H_{16}$; safrol, $C_{10}H_{10}O_2$. |
| Illicium religiosum ............. | Shikimi oil ............ | 1.006 | .... | *Terpene*, $C_{10}H_{16}$; anethol, $C_{10}H_{12}O$ (25 per cent.); safrol. |
| *Monimiaceæ:* | | | | |
| Boldoa fragrans, Gay........... | Boldo leaves oil ........ | 0.918 | 347–572 | Terpenes; oxygenous bodies. |
| Atherosperma moschata, R. Br.... | Australian sassafras oil .. | 1.04 | 435.2 | |
| *Ranunculaceæ:* | | | | |
| Nigella sativa, L. .............. | Fennel-flower oil ....... | — | — | *Terpene*, $C_{10}H_{16}$; body, $C_{20}H_{24}O$. |
| *Cruciferæ:* | | | | |
| Lepidium ruderale, L. .......... | Wall-cress oil.......... | > 1 | — | Sulphuretted. |
| Lepidium sativum, L. ........... | Garden-cress oil........ | — | 439.7 | *Phenylacetonitril*, $C_8H_7N$ (75 per cent.). |
| Lepidium latifolium, L........... | Broad-leaved pepperwort oil ................ | >1 | — | |

DERIVATION, SPECIFIC GRAVITY, BOILING POINT, AND CHEMICAL CONSTITUTION OF VOLATILE OILS.—*Continued.*

| Family, Variety. | Name of the Oil. | Specific gravity. | Boiling point, °F. | Chemical Constitution. |
|---|---|---|---|---|
| Nasturtium officinale, L. | Water-cress oil | 1.0014 | 248–536 | *Phenylpropionnitril*, $C_9H_9N$. |
| Cochlearia officinalis, L. | Scurvy-grass oil | >1 | 318.2–325.4 | *Isobutylthiocarbimide*, $C_5H_9NS$. |
| Cochlearia armoracia, L. | Horseradish oil | 1.01 | ............ | *Allylthiocarbimide*, $C_4H_5NS$. |
| Thlaspi arvense, L. | Penny-cress oil | — | — | Allylsulphide, $C_6H_{10}S$; allylthiocarbimide, $C_4H_5NS$. |
| Sinapis nigra, L. | Mustard oil | 1.006–1.012 | 298.4 | *Allylthiocarbimide*, $C_4H_5NS$; allylcyanide, $C_4H_5N$. |
| Sinapis alba, L. | White-mustard oil | ............ | ............ | *Paraoxybenzylthiocarbimide*, $C_8H_7ONS$. |
| Raphanus sativus, L. | Garden-radish oil | >1 | — | Sulphuretted. |
| Erysimum alliaria, L. | Hedge-garlic oil | — | — | Allylthiocarbimide, $C_4H_5NS$; allylsulphide, $C_6H_{10}S$. |
| *Canellaceæ:* | | | | |
| Canella alba, Murr. | Canella oil | <1 | ............ | *Eugenol*, $C_{10}H_{12}O_2$; *cineol*, $C_{10}H_{18}O$; other oxygenous bodies. |
| *Ternstroemiaceæ.* | | | | |
| Thea chinensis, Sims | Tea oil | <1 | — | |
| *Dipterocarpeæ:* | | | | |
| Dipterocarpus turbinatus | Gurjun oil | 0.918 | 491–492.8 | *Diterpene*, $C_{20}H_{32}$, or *sesquiterpene*, $C_{15}H_{24}$. |
| *Tiliaceæ:* | | | | |
| Tilia grandiflora, T. paroifolia, Ehrp. | Linden-flower oil | — | — | |
| *Geraniaceæ:* | | | | |
| Pelargonium, L'Herit | Geranium oil | 0.891–0.899 | 420.8–431.6 | *Geraniol*, $C_{10}H_{18}O$; pelargonic acid, $C_9H_{18}O_2$. |
| *Tropaeolaceæ:* | | | | |
| Tropaeolum majus, L. | Indian-cress oil | 1.0146 | 438.8 | *Phenylacetonitrile*, $C_8H_7N$. |

DERIVATION, SPECIFIC GRAVITY, BOILING POINT, AND CHEMICAL CONSTITUTION OF VOLATILE OILS.—*Continued.*

| Family, Variety. | Name of the Oil. | Specific gravity. | Boiling point, ° F. | Chemical Constitution. |
|---|---|---|---|---|
| *Rutaceæ:* | | | | |
| Ruta graveolens, L. | Rue oil | 0.84–0.91 | 338–473 | *Methyl-nonyl ketone*, $C_{11}H_{22}O$; terpene, $C_{10}H_{16}$; camphor, $C_{10}H_{18}O$. |
| Diosma betulina | Buchu leaves oil | ........... | ........... | Diosphenol, $C_{10}H_{16}O_2$. |
| Philocarpus pennatifolius | Jaborandi leaves oil | 0.875 | 352.4–554 | Limonene, $C_{10}H_{16}$. |
| Galipea officinalis, Hanc. | Angostura bark oil | 0.956 | — | |
| *Aurantiaceæ:* | | | | |
| Citrus vulgaris, Risso | Neroli oil | 0.889 | 343.4–356 | *Limonene*, $C_{16}H_{16}$ (99 per cent.). |
| Citrus vulgaris, Risso | Bitter orange peel oil | 0.83–0.86 | 347–350.6 | *Limonene*, $C_{10}H_{16}$; bodies, $C_{20}H_{30}O_3$ (2.8 per cent.); myristicol, $C_{10}H_{16}O$ (0.3 per cent.) |
| Citrus, Aurantium, Risso | Sweet orange peel oil | 0.84–0.85 | 345.2–356 | |
| Citrus Bigaradia | Mandarin oil | — | 352.4 | *Limonene*, $C_{10}H_{16}$. |
| Citrus varieties | Petits grains oil | 0.87–0.88 | — | *Limonene*, $C_{10}H_{16}$. |
| Citrus Bergamia, Risso | Bergamot oil | 0.85–0.89 | 329–374 | *Limonene*, $C_{10}H_{16}$; hydrocarbon; dipentene, $C_{10}H_{16}$; bergaptene, $C_{11}H_{10}O_4$. |
| Citrus Limetta, Risso | Lime oil | 0.88–0.93 | 338 | *Limonene*, $C_{10}H_{16}$; terpinol, $C_{10}H_{18}O$; methyl-nonyl ketone, $C_{11}H_{22}O$; diterpene, $C_{20}H_{32}$. |
| Citrus Limonum, Risso | Lemon oil | 0.85–0.87 | 350–482 | *Limonene*, $C_{10}H_{16}$; pinene, $C_{10}H_{16}$ (1.67 per cent.); cymol, $C_{10}H_{14}$; cineol, $C_{10}H_{18}O$?; citral ($C_{10}H_{16}O$?, 7.5 per cent.) |
| Citrus medica, Risso | Cedrat oil | 0.85–0.87 | — | *Terpene*, $C_{10}H_{16}$. |
| Citrus Lumia, Risso | Sweet lime oil | — | 266–356 | *Limonene*, $C_{10}H_{16}$. |
| *Polygalaceæ:* | | | | |
| Polygala Senega, L. | Senega oil | — | — | Valerianic acid ester; methyl salicylate, $C_8H_8O_3$. |

DERIVATION, SPECIFIC GRAVITY, BOILING POINT, AND CHEMICAL CONSTITUTION OF VOLATILE OILS.—*Continued.*

| Family, Variety. | Name of the Oil. | Specific gravity. | Boiling point, ° F. | Chemical Constitution. |
|---|---|---|---|---|
| *Simarubaceæ:* | | | | |
| Xanthoxylum Hamiltonianum | Evodia oil | 0.84 | — | |
| *Burseraceæ:* | | | | |
| Balsamea Myrrha, Engl. | Myrrh oil | 1.0819 | 510.8 | $C_{10}H_{14}O$ (carvol or thymol?). |
| Boswellia Carteri, Burtw. | Olibanum oil | 0.866 | 320–338 | *Levo-pinene*, $C_{10}H_{16}$; dipentene? oxygenous bodies. |
| Icica Icicariba? | Elemi oil | 0.82–0.83 | 330.8–348.8 | *Dipentene*, $C_{10}H_{16}$; *phellandrene*, $C_{10}H_{16}$; polyterpenes; oxygenous bodies. |
| { Icica altissima? | Linaloe oil | 0.87 | 392 | $C_{10}H_{18}O$? } Licareol, $C_{10}H_{18}O$? |
| { Elaphrium graveolens, Knuth? | Licaria oil | 0.868 | 388.4 | $C_{10}H_{18}O$? } |
| *Anarcadiaceæ:* | | | | |
| Pistacia Lentiscus, L. | Mastic oil | — | 311–330.8 | *Terpene*, $C_{10}H_{16}$. |
| *Vitaceæ:* | | | | |
| Vitis vinifera, L. | Wine or cognac oil | — | — | *Ethyl caprinate*, $C_{12}H_{24}O$; ethyl caprylate, $C_{10}H_{20}O_2$. |
| *Euphorbiaceæ:* | | | | |
| Croton Eluteria, Benn. | Cascarilla oil. | 0.91–0.94 | 356 | Hydrocarbons; oxygenous bodies. |
| *Umbelliferæ:* | | | | |
| Cicuta virosa, L. | Water hemlock oil | — | — | From seeds: *Cuminol*, $C_{10}H_{12}O$; cymol, $C_{10}H_{14}$. From root: *Cicutene*, $C_{10}H_{16}$. |
| Pastinaca sativa, L. | Parsnip oil | 0.87 | 471.2 | *Octyl butyrate*, $C_{12}H_{24}O_2$; propionic acid-octyl ester. |

Derivation, Specific Gravity, Boiling Point, and Chemical Constitution of Volatile Oils.—*Continued.*

| Family, Variety. | Name of the Oil. | Specific gravity. | Boiling point, °F. | Chemical Constitution. |
|---|---|---|---|---|
| Heracleum spondylium, L | Heracleum | 0.864 | 374–518 | *Octyl acetate*, $C_{10}H_{20}O_2$ (60 per cent.); other esters. |
| Heracleum giganteum, L. |  | — | 266–482 | *Hexyl butyrate*, $C_{10}H_{20}O_2$; *octyl acetate*, $C_{10}H_{20}O_2$. |
| Pimpinella anisum, L | Aniseed oil | 0.98–0.995 | 431.6–442.4 | *Anethol*, $C_{10}H_{12}O$ (80–95 per cent.); terpene? |
| Pimpinella Saxifraga, L. | Pimpinella oil | 0.959 | 464–572 |  |
| Foeniculum officinale, All. | Fennel oil | 0.94–1.00 | 374–437 | *Anethol*, $C_{10}H_{12}O$ (60–70 per cent); phellandrene; Dextro-pinene: dipentene, $C_{10}H_{16}$. |
| Phellandrium aquaticum, L | Phellandrium oil | 0.852 | — | *Phellandrene*, $C_{10}H_{16}$. |
| Anethum graveolens, L. | Dill oil | 0.89 | — | *Limonene*, $C_{10}H_{16}$ (60 per cent.); carvol $C_{10}H_{14}O$ (10 per cent). |
| Carum Carvi, L | Caraway oil | 0.88–0.97 | 379.4–388.4 | *Limonene*, $C_{10}H_{16}$ (35–55 per cent.); *carvol*, $C_{10}H_{14}O$ (45–65 per cent). |
| Carum Ajowan, Benth. | Ajowan oil | 0.896 | 320–428 | *Thymene*, $C_{10}H_{16}$ (30 to 40 per cent.); *thymol*, $C_{10}H_{14}O$; cymol, $C_{10}H_{14}$ (15–20 per cent.); carvacrol, $C_{10}H_{14}O$. |
| Cuminum Cyminum, L. | Cumin oil | 0.973 | 338–446 | *Cuminol*, $C_{11}H_{12}O$ (77 per cent.); cymol, $C_{10}H_{14}$ (23 per cent). |
| Petroselinum sativum, L. | Parsley oil | 1.052 | 320–554 | *Terpene*, $C_{10}H_{16}$; apiol, $C_{12}H_{14}O_4$. |
| Athamanta Oreoselinum, Mönch | Athamanta oil | 0.843 | 325.4 | *Terpene*, $C_{10}H_{16}$. |
| Apium graveolens, L. | Celery oil | 0.881 | — |  |
| Angelica Archangelica, L. | Angelica oil | from the root:<br>0.875 | 320–347 | *Terpene*, $C_{10}H_{16}$ (75 per cent.); polyterpenes. |
| Angelica Archangelica, L. |  | from seed:<br>0.855 | 348.8–536 | *Terpene*, $C_{10}H_{16}$; methyl-ethyl acetic acid, $C_5H_{10}O_2$; oxymyristic acid, $C_{14}H_{28}O_3$. |
| Levisticum officinale, Koch | Lovage oil | 0.935 | — |  |

DERIVATION, SPECIFIC GRAVITY, BOILING POINT, AND CHEMICAL CONSTITUTION OF VOLATILE OILS.—*Continued.*

| Family, Variety. | Name of the Oil. | Specific gravity. | Boiling point, ° F. | Chemical Constitution. |
|---|---|---|---|---|
| Ferula Sumbul, Hook fil. | Sumbul root oil | 0.954 | — | Angelic acid, $C_5H_8O_2$; valerianic acid, $C_5H_{10}O_2$; umbelliferon, $C_2H_6O_3$? |
| Dorema Ammoniacum, Don. | Ammoniacum oil | 0.891 | 482–554 | |
| Ferula Narthex, Boiss. | Asafoetida oil | 0.915 | 275 | *Allyl sulphide*, $C_6H_{10}S$; allyldisulphide, $C_6H_{10}S_2$. Blue oil. |
| Ferula galbaniflua, Boiss. | Galbanum oil | 0.84–0.96 | 321.8–338 | *Dextro-pinene*, $C_{10}H_{16}$; sesquiterpene, $C_{15}H_{24}$. |
| Ferula Opoponax, L. | Opoponax oil | 0.901 | 392–572 | |
| Daucus Carota, L. | Carrot oil | from the root: 0.886 | | |
| Daucus Carota, L. | Carrot oil | from fruits: 0.883 | 212–464 | *Dextro-pinene*, $C_{10}H_{16}$, $C_{10}H_{18}O$ (40 per cent.); cineol? |
| Imperatoria Ostruthium, L. | Masterwort oil | 0.877 | 338–428 | Hydrocarbon; terpene hydrates; angelic aldehyde, $C_5H_8O$? |
| Coriandrum sativum, L. | Coriander oil | 0.871–0.872 | 302–392 | *Coriandrol*, $C_{10}H_{18}O$. |
| *Saxifragaceæ:* | | | | |
| Liquidambar orientale, Mill. | Storax oil | — | — | Styrol, $C_8H_9$; cinnamic acid ester. |
| *Myrtaceæ:* | | | | |
| Melaleuca Leucadendron | Cajeput oil | 0.89–0.98 | 347–485.6 | *Cineol*, $C_{10}H_{18}O$ (67 per cent.); terpene, $C_{10}H_{16}$; terpilenol, $C_{10}H_{18}O$; aldehyde, ester, polyterpenes. |
| Myrtus Cheken | Cheken leaves oil | — | — | *Pinene*, $C_{10}H_{16}$ (75 per cent.); cineol, $C_{10}H_{18}O$ (25 per cent.) |
| Myrtus Pimenta, L.<br>Myrtus acris, etc. | Pimento oil | 1.03–1.05 | — | *Sesquiterpene*, $C_{15}H_{24}$ (30 to 67 per cent.); *eugenol*, $C_{10}H_{12}O_2$ (33 to 70 per cent.). |
| Pimenta acris, Wight | Myrcia or bay oil | 0.97–0.98 | 320–482 | *Pinene, dipentene*, $C_{10}H_{16}$ (60 to 70 per cent.); eugenol, $C_{10}H_{12}O_2$ (30 to 40 per cent.). |

DERIVATION, SPECIFIC GRAVITY, BOILING POINT, AND CHEMICAL CONSTITUTION OF VOLATILE OILS.—*Continued.*

| Family, Variety. | Name of the Oil. | Specific gravity. | Boiling point, °F. | Chemical Constitution. |
|---|---|---|---|---|
| Myrtus communis, L. | Myrtle oil | 0.91 | 320–464 | Dextro-pinene, dipentene, $C_{10}H_{16}$; cineol, $C_{10}H_{18}O$; camphor. |
| Eucalyptus globulus, Labill | Common eucalyptus oil | 0.904–0.921 | 338–374 | *Cineol*, $C_{10}H_{18}O$ (50 to 70 per cent.); dextropinene, $C_{10}H_{16}$; fatty acid aldehyde. |
| Eucalyptus amygdalina, L. | Australian eucalyptus oil | 0.874–0.897 | 318.2-482 | *Phellandrene*, $C_{10}H_{16}$; cineol, $C_{10}H_{18}O$. |
| Eucalyptus maculata var. citriodora, Hérit | Lemon-scented gum-tree oil | 0.873–0.905 | 408.2–428 | *Citronellon* ($C_{10}H_{18}O$?, 75 per cent.). |
| Eucalyptus Backhousia | — | 0.9 | 433.4–451.4 | *Citral*, $C_{10}H_{16}O$. |
| Carophyllus aromaticus | Clove oil | 1.04–1.07 | 476.6 | *Eugenol*, $C_{10}H_{12}O_2$ (90 per cent.); sesquiterpene, $C_{15}H_{24}$. |
| Carophyllus aromaticus | Clove stalks oil | 1.009–1.063 | — | *Eugenol*, $C_{10}H_{12}O_2$; sesquiterpene, $C_{15}H_{24}$. |
| *Rosaceæ:* | | | | |
| Rosa varieties | Rose oil | 0.83–0.89 | 444.2 | *Elaeoptene* (66 to 88 per cent.); stearoptene (12 to 34 per cent., mixture of hydrocarbons). |
| Spiraea ulmaria, L. | Spiraea oil | — | — | *Salicylaldehyde*, $C_7H_6O_2$; terpene, $C_{10}H_{16}$; camphor. |
| *Drupaceæ:* | | | | |
| Amygdalis communis, L. var amara. | Bitter almond oil | 1.04–1.07 | 356 | *Benzaldehyde*, $C_7H_6O$ (76 per cent.); mandelic acid nitrile, $C_8H_7NO$ (24 per cent.?). |
| Prunus Laurocerasus, L. | Cherry laurel oil | — | — | Same as bitter almond oil. |
| Prunus virginiana | Wild cherry bark oil | 1.05 | — | *Benzaldehyde*, $C_7H_6O$; prussic acid, HCN. |
| *Papilionaceæ:* | | | | |
| Myroxylon Pereira, Baill. | *a.* Peru balsam oil | 1.12 | — | *a.* Benzyl-benzoate, $C_{14}H_{12}O_2$; benzyl-cinnamate, $C_{16}H_{14}O_2$; styracin, $C_{18}H_{16}O_2$. |
| | *b.* the same, extracted | — | — | *b.* *Benzyl-cinnamate*, $C_{16}H_{14}O_2$. |
| | *c.* oil from the leaves | 0.874 | — | |
| | *d.* oil from the wood | 0.892 | — | |

DERIVATION, SPECIFIC GRAVITY, BOILING POINT, AND CHEMICAL CONSTITUTION OF VOLATILE OILS.—*Continued.*

| Family, Variety. | Name of the Oil. | Specific gravity. | Boiling point, °F. | Chemical Constitution. |
|---|---|---|---|---|
| Toluifera Balsanum, L. .......... | Tolu balsam oil ........ | — | — | Cinnamein, $C_{10}H_{14}O_2$; benzoic acid, $C_7H_6O_2$; toluene, $C_{10}H_{16}$. |
| *Cæsalpiniaceæ:* | | | | |
| Copaifera varieties.............. | Copaiva oil............ | 0.88–0.96 | 473–527 | *Diterpene*, $C_{20}H_{32}$; diterpene hydrate, $C_{80}H_{98}O$; products of oxidation. |
| *Ericaceæ:* | | | | |
| Gaultheria procumbens, L........ | Wintergreen oil ........ | 1.18–1.1835 | 392–431.6 | *Methylsalicylate*, $C_8H_8O_3$ (99.7 per cent.); gaultherilen, $C_{10}H_{16}$ (0.3 per cent). |
| Andromeda Leschenaultii........ | Andromeda oil. | | | |
| Ledum palustre ................ | Marsh tea oil .......... | — | — | *Ledum camphor*, $C_{15}H_{26}O$; sesquiterpene, $C_{15}H_{24}$. |
| *Oleaceæ:* | | | | |
| Syringa vulgaris, L.............. | Lilac oil. | | | |
| *Convolvulaceæ:* | | | | |
| Convolvulus scoparius, L ........ | Rosewood oil .......... | 0.9064 | — | *Terpene*, $C_{10}H_{16}$ (80 per cent). |
| *Labiatæ:* | | | | |
| Thymus vulgaris................ | Thyme oil............. | 0.87–0.94 | 338–356 | *Thymene*, $C_{10}H_{16}$; thymol, $C_{10}H_{14}O$ (15 per cent.); cymol, $C_{10}H_{14}$. |
| Monarda punctata, L............ | Horsemint oil.......... | — | — | *Terpene*, $C_{10}H_{16}$ (50 per cent.); thymol, $C_{10}H_{14}O$ (20–30 per cent; products of oxidation; camphor; ester. |
| Thymus serpyllum, L............ | Wild thyme oil......... | 0.89–0.92 | .... | *Cymol*, $C_{10}H_{14}$; *thymol*, $C_{10}H_{14}O$; carvacrol, $C_{10}H_{14}O$; terpene, $C_{10}H_{16}$? |

DERIVATION, SPECIFIC GRAVITY, BOILING POINT, AND CHEMICAL CONSTITUTION OF VOLATILE OILS.—*Continued.*

| Family, Variety. | Name of the Oil. | Specific gravity. | Boiling Point, ° F. | Chemical Constitution. |
|---|---|---|---|---|
| Mentha piperita, L. | Peppermint oil | 0.84–0.96 | 372.2–413.6 | *Menthene*, $C_{10}H_{18}$ (45–80 per cent.); *menthol*, $C_{10}H_{20}O$ (20 to 55 per cent.); aldehyde; ketone; menthone, $C_{10}H_{18}O$. |
| Mentha viridis, L. | Spearmint oil | 0.9142 | .... | *Carvol*, $C_{10}H_{14}O$; menthene, $C_{10}H_{16}$; resin. |
| Mentha crispa, L. | Crisped mint oil | 0.93–0.98 | 392–446 | *Carvol*, $C_{10}H_{14}O$ (25–50 per cent.). |
| Mentha Pulegium, L. | European pennyroyal oil. | 0.93–0.94 | 359.6–446 | Pulegon. |
| Hedeoma pulegioides, Pers. | American pennyroyal oil. | 0.93–0.94 | 302–536 | Pulegon. |
| Melissa officinalis, L. | Melissa or balm oil | 0.85–0.98 | | |
| Lavandula vera, D. C. | Lavender oil | 0.87–0.94 | 366.8–377.6 | *Camphor* (65 per cent.); terpene, $C_{10}H_{16}$ (25 per cent.); resin (10 per cent.). |
| Lavandula spica, D. C. | Spike lavender oil | 0.87–0.96 | 330.8–366.8 | *Terpene*, $C_{10}H_{16}$ (35–70 per cent.); *cineol*, *camphor* (20–55 per cent.); resin (10 per cent.). |
| Rosmarinus officinalis, L. | Rosemary oil | 0.88–0.92 | 302–500 | *Levo-pinene*, $C_{10}H_{16}$ (80 per cent.); borneol, $C_{10}H_{18}O$; camphor, $C_{10}H_{16}O$ (9.5–12.4 per cent.); cineol, $C_{10}H_{18}O$. |
| Ocymum Basilicum, L. | Sweet basil oil | — | — | Basil camphor, $C_{10}H_{22}O_3$. |
| Pogostemon Patchouly, Pell. | Patchouli oil | 0.95–1.01 | 539.6–561.2 | *Sesquiterpene*, $C_{15}H_{24}$; *patchouli camphor*, $C_{15}H_{26}O$. |
| Salvia officinalis, L. | Sage oil | 0.86–0.92 | 275–583 | *Salviol*, $C_{10}H_{16}O$; pinene, $C_{10}H_{16}$; terpene, $C_{10}H_{16}$; cymol, $C_{10}H_{14}$; cineol, $C_{10}H_{18}O$; sage camphor, $C_{10}H_{16}$; sesquiterpene, $C_{15}H_{24}$. |
| Hyssopus officinalis, L. | Hyssop oil | 0.89–0.99 | 287.6–325.4 | |
| Origanum vulgare, L. | Origanum oil | 0.87–0.91 | 321.8 | *Camphor*; terpene; phenol (0.1 per cent.). |
| Origanum hirtum, Koch. | Cretic origanum oil | 0.95–0.98 | — | *Carvacrol*, $C_{10}H_{14}O$ (50–80 per cent.); terpene, $C_{10}H_{16}$; cymol, $C_{10}H_{14}$. |
| Origanum Majorana, L. | Sweet marjoram oil | 0.89–0.92 | 325.4 | *Sesquiterpene hydrate*, $C_{15}H_{26}O$; terpene, $C_{10}H_{16}$. |

DERIVATION, SPECIFIC GRAVITY, BOILING POINT, AND CHEMICAL CONSTITUTION OF VOLATILE OILS.—*Continued.*

| Family, Variety. | Name of the oil. | Specific Gravity. | Boiling point, ° F. | Chemical Constitution. |
|---|---|---|---|---|
| Satureja hortensis, L. | Savory oil | 0.898 | — | *Terpene*, $C_{10}H_{16}$ (50 per cent); carvacrol, $C_{10}H_{14}O$ (30 per cent.); cymol, $C_{10}H_{14}$ 20 per cent.). |
| Satureja montana |  | 0.7394 | — | *Carvacrol*, $C_{10}H_{14}O$ (35–40 per cent.); phenol; terpenes. |
| *Verbenaceæ:* |  |  |  |  |
| Verbena triphylla, Hérit. | Verbena oil | 0.89–0.896 | 429.8 |  |
| *Caprifoliaceæ:* |  |  |  |  |
| Sambucus nigra, L. | Elder flower oil | ........... | ........... | Terpene, $C_{10}H_{16}$. |
| *Valerianaceæ:* |  |  |  |  |
| Valeriana officinalis, L. | *a.* Valerian oil | 0.94–0.97 | 248–752 | *Alcohol*, $C_{10}H_{18}O$; *bornyl ester; bornyl ether*, $C_{20}H_{34}O$; pinene, $C_{10}H_{16}$ (25 per cent.); valerianic acid, $C_5H_{10}O_2$ (5 per cent.). |
| Valeriana var. angustifolia | *b.* Kesso root oil | 0.996 | 338–581 | *Levo-borneol*, $C_{10}H_{18}O$; *terpineol*, $C_{10}H_{18}O$; *bornyl ester; kessyl acetate*, $C_{16}H_{26}O_3$; aldehyde; fatty acid; pinene; dipentene; sesquiterpene? blue oil. |
| *Compositæ:* |  |  |  |  |
| Anthemis nobilis, L. | Roman chamomile oil | — | 320–410 | *Isobutyl angelicate*, $C_9H_{16}O_2$; *amyl angelicate*, $C_{10}H_{18}O_2$; *amyl tiglinate*, $C_{10}H_{18}O_2$; hydrocarbon; ester; anthemol, $C_{16}H_{16}O$; angelic acid, $C_5H_8O_2$? |
| Matricaria Chamomilla, L. | Chamomile oil | 0.92–0.95 | 221–572 | *Camillol*, $C_{10}H_{16}O$; terpene, $C_{10}H_{16}$; blue oil. |
| Matricaria Parthenium, L. | Feverfew oil | 0.89–0.97 | 302–428 | *Matricaria camphor*, $C_{10}H_{16}O$; terpene, $C_{10}H_{16}$. |
| Artemisia maritima, L. | Levant wormseed oil | 0.93 | 347–428 | *Cineol*, $C_{10}H_{18}O$; terpene, $C_{10}H_{16}$; propionate or acetone; oxygenous bodies. |
| Artemisia vulgaris, L. | Mugwort oil. |  |  |  |

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DERIVATION, SPECIFIC GRAVITY, BOILING POINT, AND CHEMICAL CONSTITUTION OF VOLATILE OILS.—*Continued.*

| Family, Variety. | Name of the Oil. | Specific gravity. | Boiling point, °F. | Chemical Constitution. |
|---|---|---|---|---|
| Artemisia glacialis, Luc.......... | Alpine mugwort oil..... | 0.964 | 383–590 | |
| Artemisia Dracunculus, L........ | Tarragon oil........... | 0.94–0.95 | 392–402.8 | *Anethol*, $C_{10}H_{12}O$; hydrocarbon. |
| Artemisia Absinthium, L ........ | Wormwood oil.......... | 0.88–0.94 | 356–401 | *Absinthol*, $C_{10}H_{16}O$; terpene; hydrocarbon; blue oil. |
| Achillea Millefolium, L.......... | Milfoil oil .............. | 0.85–0.92 | — | |
| Achillea nobilis, L.............. | Yarrow or milfoil oil.... | 0.97–0.98 | | |
| Achillea moschata, D. C......... | Iva oil ................ | 0.934 | 338–500 | *Ivaol*, $C_{12}H_{20}O_3$; resin. |
| Achillea ageratum, L............ | ........................ | 0.849 | 329–359.6 | Bodies, $C_{28}H_{44}O_3$. |
| Erigeron canadense, L .......... | Erigeron oil ............ | 0.85–0.87 | 348.8 | *Limonene*, $C_{10}H_{16}$; resin. |
| Tanacetum vulgare, L........... | Tansy oil............... | 0.923 | 377.6–536 | *Tanacetol*, $C_{10}H_{16}O$ (70 per cent.); tanacetyl alcohol, $C_{10}H_{18}O$ (26 per cent.); terpene, $C_{10}H_{16}$ (1 per cent.); resin; malic acid, $C_4H_6O_5$? |
| Arnica montana, L ............. | *a*. Arnica flower oil..... | 0.9 | | |
| | *b*. Arnica root oil....... | 0.98–1.01 | 417.2–505.4 | *Thymohydroquinone methyl ether* $C_{12}H_{18}O_2$ (80 per cent.); phloryl butyrate, $C_{12}H_{16}O_2$ (20 per cent.); phlorol methyl ether, $C_9H_{12}O$. |
| Inula Helenium, L.............. | Elecampane oil ........ | — | — | *Alantol*, $C_{10}H_{16}O$; *alant acid anhydride*, $C_{15}H_{20}O_2$. |
| Carlina acaulis, L.............. | Carline thistle oil....... | 1.03 | 509–572 | *Sesquiterpene*, $C_{15}H_{24}$ (12 per cent). |
| *Santalaceæ:* | | | | |
| Santalum album, L ............. | Sandal wood oil........ | 0.95–0.98 | 410–644 | *Santalal*, $C_{15}H_{24}O$; santalol, $C_{15}H_{26}O$. |
| *Aristolochiaceæ:* | | | | |
| Aristolochia Clematitis, L........ | Aristolochy oil.......... | 0.9 | | |
| Aristolochia Serpentaria, Jacq.... | Snakeroot oil. | | | |

DERIVATION, SPECIFIC GRAVITY, BOILING POINT, AND CHEMICAL CONSTITUTION OF VOLATILE OILS.—*Concluded.*

| Family, Variety. | Name of the Oil. | Specific gravity. | Boiling point, ° F. | Chemical Constitution. |
|---|---|---|---|---|
| Asarum Europæum, L. | Asarum oil | 1.046 | 338–572 | *Methyl eugenol*, $C_{11}H_{14}O_2$; Levo-pinene, $C_{10}H_{16}$ (4.5 per cent.); asaron, $C_{12}H_{16}O_3$. |
| Asarum Canadense | Wild ginger oil | .... | .... | Asarol, $C_{10}H_{18}O$; fatty acid and aserol ester; asarene, $C_{10}H_{16}$. |
| *Family unknown:* | | | | |
| Meum athamanticum, Jacq | Mew oil | 0.999 | 338–572 | |
| Turnera | Damiana leaf oil | — | 482–590 | Blue oil. |
| ? | Dilem leaf oil | 0.96 | 482–572 | |
| Erechthitis hiearacifolia | Erechthitis oil | 0.85–0.86 | 354.2–374 | Terpenes, $C_{10}H_{16}$; sesquiterpene, $C_{15}H_{24}$. |
| ? | Musk seed oil | 0.9 | | |
| Osmitopsis asteriscoides | Osmitopsis oil | 0.931 | 348.8–406.4 | Cineol, $C_{10}H_{18}O$. |

# CHAPTER XX.

## MANNER OF OBTAINING VOLATILE OILS.

THE methods employed for the purpose of obtaining volatile oils differ very much, according to the condition of the part of the plant and the quality of oil contained in it. It is self-evident that, to obtain the odoriferous principle from a small plant, a different method will have to be employed than for the preparation of volatile oil from wood or fleshy fruit.

Whatever method is to be used depends, however, not so much on the condition of the part of the plant, but chiefly on the properties of the volatile oil occurring in it. As an example, we would only mention orange peel, which retains the quality of its odor, even when shriveled up and leathery, losing only a portion of its oil by evaporation and resinification.

Rose leaves dried in the air retain for years an odor faintly recalling that of the rose, but the odor of violets and other flowers disappears entirely before the plant is even completely wilted, and is replaced by the characteristic smell all plants acquire in drying.

The manufacturer, wishing to prepare volatile oils from such plants, has therefore to cultivate them himself, or make arrangements to receive sufficient quantities of them in a perfectly fresh condition. The work of the factory should be so arranged that, during the flowering period of the plants, all resources can be employed to work up as large a quantity as possible of this perishable raw material.

The principal seats of the preparation of volatile oils from fresh flowers are Grasse, Cannes, and Nice, and also Monaco. The climate of these places enables the grower to bring the plants to the highest perfection. Many hundreds of acres of the best soil are planted with violets, jasmine, accacia, etc., solely for the purpose of furnishing flowers for the manufacture of volatile oils.

In England and America the production of volatile oil is generally restricted to that of lavender, peppermint, sassafras, wintergreen, birch, and a few others, though there can be no doubt, as previously stated, that many northern as well as southern districts of the United States are well adapted for the cultivation of quite a number of plants grown to advantage in Southern Europe, which might be made to yield highly valuable articles of commerce.

As for the processes in use for extracting the volatile oils from plants, there are six: Expression; distillation; extraction with a solvent at an ordinary temperature, either with or without pressure; maceration or infusion; absorption; and absorption with the aid of hot air.

Which of these processes is to be used depends entirely on the condition of the raw material; for many, several of the methods named may be advantageously employed, while for others only one method is available for obtaining a product of good quality.

Some raw materials require special preparation to obtain the oil contained in them. This is chiefly the case with woods which have to be comminuted by suitable mechanical contrivances. Woods are used either in the form of thin shavings, or, better, as fine sawdust. Their conversion into shavings is effected either by hand with a plane, or by means of a planing machine. A simple but effective construction of the latter consists of an iron drum, provided on its periphery with three to six knife blades, set obliquely, and revolving rapidly. The wood to be planed is placed upon a carriage which conducts it against the knives in such a manner that it is cut in a direction running horizontal to the longitudinal fibres, whereby crummy shavings are secured.

For the conversion of the wood into sawdust a drum of sheet steel is used. The surface of this drum forms a rasp against which the wood to be comminuted is pressed. To prevent the cuts of the rasp from being clogged up with sawdust, the latter is constantly rinsed off by a jet of water falling upon the drum.

Nuts and barks are crushed by fluted rolls, and roots and woody herbs comminuted by means of a suitable cutter.

Hard seeds, such as caraway, anise, etc., are comminuted by crusher rolls, for description of which we refer to Fixed Oils, Vol. 1, page 240, *et seq.* As in many cases the volatile oil occurs only in the shells or husks, care must be had to see that they are thoroughly torn asunder.

The steam-boiler of a factory for the manufacture of volatile oils should be sufficiently large to furnish steam for the engine and for distillation, etc. It should be so located that the steam is available in all parts of the factory, as it frequently becomes necessary to heat the air in some rooms.

In factories which prepare all sorts of volatile oils, large rooms for storing raw material are required. They should be perfectly dry, and the stored material must not show the slightest sign of mould, as that would affect the quality and the odor of the volatile oil prepared from them. The cellar for storing the volatile oils should have as low a temperature as possible, to prevent the oils from undergoing any material change.

We will now consider the different processes of extracting the oils.

### 1. *Process of Expression.*

This is only practicable when the substances are especially rich in oil and of sufficient softness, as is the case with the peel of the orange, citron, lemon, etc. In such instances the material is simply placed in a linen cloth and subjected to a strong pressure until it ceases to yield oil. The press may be of any size, according to the quantity to be expressed. For small quantities it generally consists of an iron vessel, having a small opening at the bottom so that the oil may flow out. The material is placed upon a perforated bottom inside of the vessel and covered with a well-fitting iron plate, that can be pressed down by means of a screw. Though the material is fairly exhausted by such a press, for large operations it is advisable to make use of a hydraulic press, which is constructed and managed in exactly the same manner as those used for the expression of fixed oils.

By expression a turbid milky fluid is obtained, which consists of the volatile oil and aqueous substances. The latter are a solution of various extractive substances and salts in water. This fluid, as it runs from the press, is received in tall, narrow, glass vessels and brought into a cool place for clarification. This frequently requires several days, three layers being generally distinctively distinguished. On the bottom is a slimy layer consisting of cell substance carried along by the liquid bodies. Over this is a clear fluid consisting of a solution of extractive substances, vegetable albumen, and salts, and upon this floats the volatile oil, being specifically the lighter body, which, by its greater refractive power, can be clearly distinguished from the aqueous fluid.

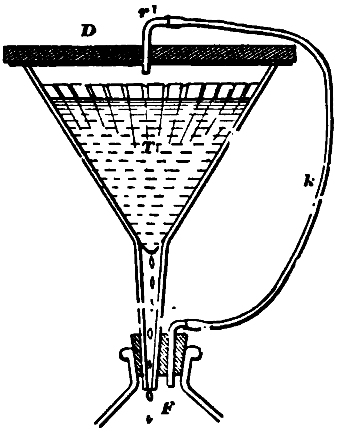
The oil is separated by bringing all that has been expressed into a bottle provided near the bottom with a lateral neck closed by a cock. After separating the oil from the aqueous fluid, the latter is allowed to escape by opening the cock.

The oils obtained in this manner are still impure, and require further treatment to remove small vegetable fibres, invisible to the naked eye, floating in them, which cause them to be somewhat opaque and slightly opalescent, and by their subsequent decomposition would give the oils a disagreeable odor.

There are two methods of obtaining the oils entirely clear, viz., filtration and distillation. Filtration is the cheaper process, but requires special precautions to exclude the air as much as possible to prevent the oil from undergoing injurious changes. By arranging the filtering apparatus so that the oil always comes only in contact with the same quantity of air, the injurious action of the oxygen is reduced to a minimum. It is self-evident that the apparatus should not be placed in the sun, but in a semi-dark, cool place.

A filter of simple construction and performing excellent service is shown in Fig. 268. It consists of a large glass bottle *F*, hermetically closed by a cork with two holes. The neck of the glass funnel *T*, the upper rim of which is ground smooth, is placed in one of the holes, and a glass tube *r*, bent at a right angle, is fitted into the second hole. A thick wooden lid, with a rubber ring on the lower side, is placed upon the funnel, thus closing it air tight. In the centre of the lid is fitted a glass tube *r′*, also bent at a right angle, which is connected with the tube *r* by a rubber hose *k*. After the funnel has been provided with filtering paper and the oil to be filtered, the lid is placed upon it, and must not be removed except for the purpose of pouring more oil into the funnel. The air in the bottle *F* is displaced by the oil dropping into it, and escapes through *r*, *k*, and *r′* into the funnel, and thus only the air in the bottle and funnel can act upon the oil.

FIG. 268.

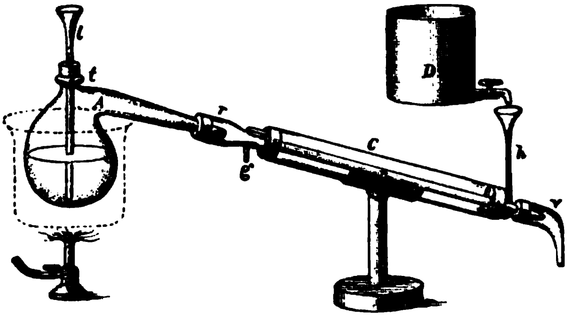


The other method for the complete purification of expressed oils is by rectification or distillation with water. For this purpose the oil, together with a little water, is brought into one of the stills described later on and the oil distilled over. It is somewhat difficult to obtain the last portion of the oil, especially with a still heated by direct fire, and it is therefore preferable to combine it with a fresh quantity of oil to be distilled.

### 2. *Process of Distillation.*

There are at present two methods in use. The one is founded upon the direct action of heat, the other upon the use of steam. The first was formerly in general practice, and is still largely employed in France and England, and to a limited extent in this country. It is, however, very deficient in many respects. As the stills must necessarily be of small capacity, only small quantities can be distilled at one time, and the oils very rarely possess the peculiar color due to them, and sometimes the odor is even altered. In mixing too little water with the material to be extracted there is danger that empyreumatic oils will be formed; a great quantity of water, on the other hand, is of disadvantage, in so far as in case the aromatic substance contains little oil, only an odoriferous water, but no oil, will be obtained. In order to avoid these inconveniences, or at least to do away with some of them, another plan was devised The material to be distilled was spread upon sieves, which were suspended in the upper part of a still, so that they might be penetrated by the vapors from below. It is true that no scorching is possible in such a case, as it was in the other process when the heating was continued after all the water had evaporated; the oil retains its proper color, but then by this method only small quantities can be extracted at a time. The still generally used for distillation with direct heat resembles so much an ordinary whiskey still as to need no further description here.

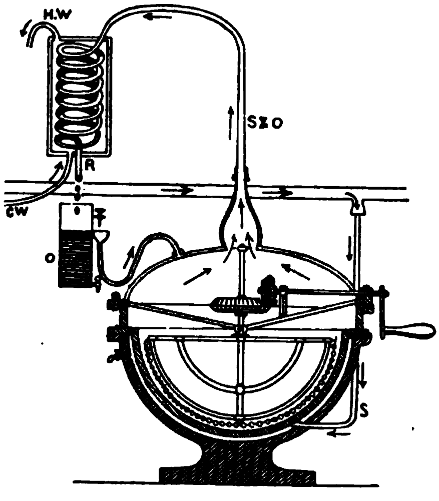
FIG. 269.



For the accurate determination of the percentage of volatile oil a vegetable substance will yield, or to obtain the oil from very costly raw material, the small glass apparatus, Fig. 269, is used. The retort *A*, with a capacity up to five or six quarts, serves for a still. In the tube *t*, shaped like the neck of a bottle, is inserted, by means of a cork, a funnel pipe *l*, reaching to the bottom of the retort. The neck of the retort passes into the cooling pipe, which lies in a so-called Liebig cooler. This consists of a wide glass tube *C*, into the lower end of which, at *k*, flows cold water from the reservoir *D*, displacing the heated water at *g*. The lower end of the cooling pipe is connected with the neck-shaped tube *v*, under which stands the vessel for the reception of the distillate. To prevent the retort from cracking, which might readily happen with the use of direct heat, it is placed in a vessel filled with sand or water.

A very good small apparatus for the distillation of volatile oil is shown in Fig. 270. It is known as a siphon still. It consists of a double-walled boiler, surmounted by a still-head, which is provided with a mechanism for keeping the contents of the boiler in motion. This stirring apparatus consists of a perpendicular shaft bearing a framework of iron curved so as to correspond to the interior shape of the still, and on the outside carrying a chain which scrapes over the inner surface of

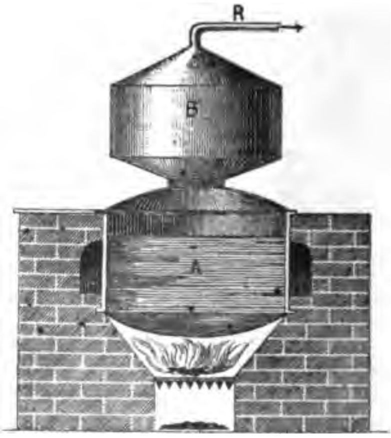
FIG. 270.



the still while the stirrer is being turned. This may be done either by hand or by steam. The still, having been charged with the material to be extracted, is filled up with water to within a few inches of the top of the body of the still, and the latter is heated by admitting steam through the pipe *S*. The vapors arising are conducted to a cooler situated at a higher level than the still itself, and the condensed liquid is collected in a receiver, where the oil and water separate. This receiver is provided with two faucets, one near the top and the other at the bottom. If the oil passing over is heavier than water, the excess of the latter is removed by the upper faucet; if the oil swims on the water, the lower faucet is regulated so as to allow the water to escape in about the same ratio as it enters the receiver. In either case the condensed water is made to run back into the still and the loss of oil is, therefore, greatly reduced.

A simple way of converting an ordinary still into use with steam is shown in Fig. 271. For the helmet of the still *A* is substituted a cylindrical vessel *B*, with an opening in the bottom. The materials to be distilled are brought into *B* and rest upon a wire bottom to prevent particles from falling into *A*.

FIG. 271.

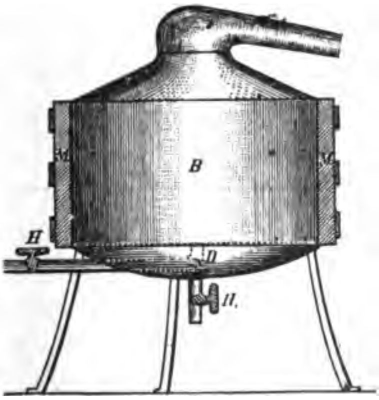


From the upper portion of *B* a pipe *R* leads to the condenser. As may be seen from the construction, the still *A* serves only for the generation of steam. The latter, in passing through *B*, heats the contents and absorbs the liberated oil, the combined vapors passing into the condenser.

This simple modification of the ordinary still affords some advantages, the principal being the avoidance of the condensation of a large quantity of water. This in itself would not amount to much, but it has to be taken into consideration that, though volatile oils are only very sparingly soluble in water, they are nevertheless soluble in it to such a degree as to impart to it their characteristic odor and taste. Such aromatized water can be utilized in the manufacture of liqueurs and perfumeries, but to the manufacturer who restricts himself to the production of volatile oils alone this represents a loss, and it is therefore necessary to him to condense as little water as possible. And this object can only be attained by the use of direct steam.

A simple apparatus for the purpose is shown in Fig. 272.

FIG. 272.

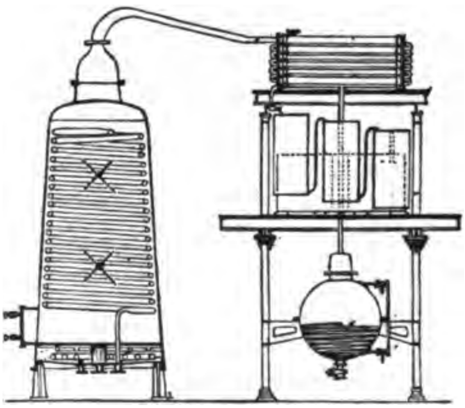


The still *B*, provided with a helmet, rests free upon a suitable support. To prevent cooling it is surrounded with a wooden jacket *M*. The material to be extracted rests upon a perforated bottom, beneath which enters the pipe *H D*, which conducts the steam from the boiler. For the more uniform distribution of the steam it is recommended to let this pipe end in a perforated coil. The water condensed in the apparatus itself is discharged through the short pipe *H*, placed in the lowest part of the still.

The size of the still varies according to requirements, though it is seldom over seven feet in diameter.

An improved apparatus for distilling volatile oils has been patented in Germany by Messrs. Schimmel & Co., of Leipzig.* The tall conical column at the left (Fig. 273) is the still. About eight inches from its bottom is a perforated diaphragm or false bottom, upon which the material to be distilled is placed by introducing it through the still-head. A perforated coil below the diaphragm projects steam upwards through the mass, which is occasionally agitated from without by means of a horizontal stirring apparatus indicated by the two crosses. Any condensed water which may run back is converted into steam by the heating coil at the bottom. Meanwhile the mass itself is heated by a long coil lining the body of the still and carrying steam at a high pressure. Whatever of volatile oil is

FIG. 273.



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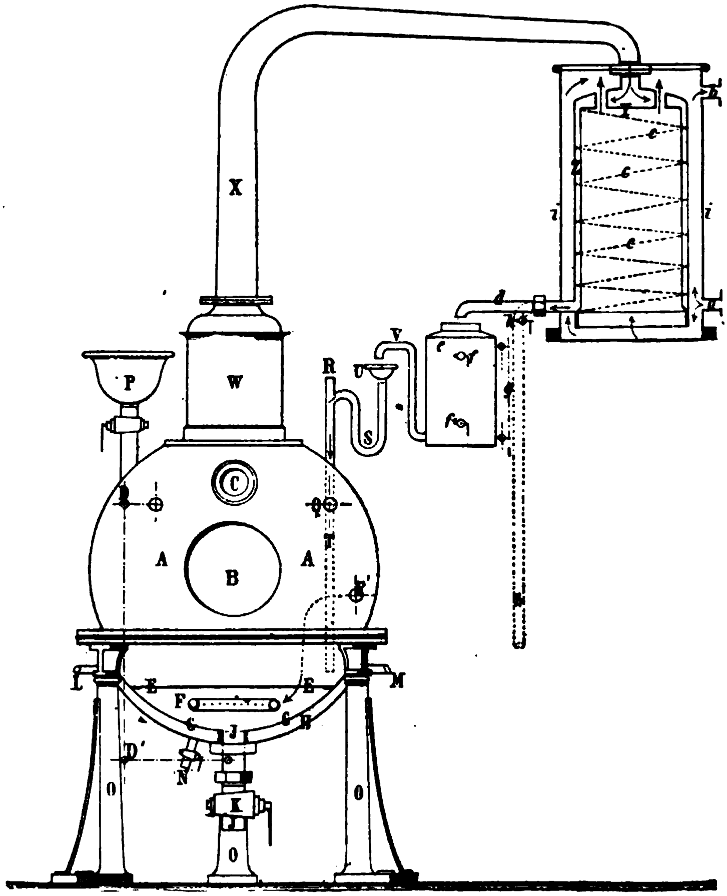
* Dingler's Polyt. Jour., Vol. 238, p. 428.

carried forward by the steam, passes through the still-head into the cooler on the right, where both oil and steam are condensed, and from whence they flow through a small funnel tube into three successive receivers, which are arranged like Florence flasks, and which retain the volatile oil that has separated. From the last receiver the water, which is still impregnated with oil, enters another reservoir, shown in the illustration only by dots, and from there it flows into a small globular still situated underneath, in which, by means of steam, nearly all the oil still retained, is again volatilized with the steam of water and both again conducted to the cooler.

Fig. 274 exhibits a smaller steam distilling apparatus with reflux condenser manufactured by Volkmar, Hänig & Co. of Dresden. The lower portion of the spherical copper still is provided, between *G* and *H*, with a steam-jacket which is heated with steam from *L*, while *M* serves as air-cock for the jacket and *N* for discharging the water of condensation. The still contains the perforated bottom *E*, below which is placed the perforated circular pipe *F* for the admission of steam into the interior of the still. The steam enters from *F*, through a pipe bent in the direction of the arrow. The still is further provided with a man-hole for introducing the plants, and a looking-hole *C* for watching the progress of distillation. During distillation the man-hole is hermetically closed. The water-gauge *DD'* passes above into the still and below, above the cock *K*, into the discharge pipe *I* of the still. At *Q* the still is provided with an air-valve; behind it lies the inlet-pipe *T* for the water of the distillate running off from the Florentine flask *e* through *VUS*. At *R* in *T* a thermometer may be inserted. The funnel *P* for filling the still rests in a collar supported by three standards *O*. On top of the still is the dome *W* with the retort-neck *X*, the latter being connected with the jacketed condenser *YZ*. In this condenser the vapors pass spirally downward in the jacket, while from *a* cold water enters the cooling vat *i*, as well as the interior of the condenser *Z*, and leaves *i* at *b*. The condensed distillate runs through *d* into the receiver *e* which, in the illustration, is drawn as used for oils specifically lighter than water. The flask is provided at different levels with two discharge-cocks *f*, and a gauge *g*. Through *V* the water runs to the

FIG. 274.



funnel *U*, and then through the pipe *S* to the pipe *T*, which extends to a short distance above the perforated bottom *E* in the still. The pipe *S* is bent ω-shaped so as to form a hydraulic trap. When distillation is finished and the water running from *V* is odorless, the distillate is discharged through *h* and the cock on $F''$ closed, while for the purpose of evaporating the water retained in *A* the jacket may be further heated. By placing a valve in *X* and keeping it closed steam under increased pressure may, in the beginning of distillation, be allowed to act upon the plants whereby the bursting of the oil-bearing cells is in many cases facilitated. It may finally be remarked that the apparatus may also be used for rectifying the oil, as well as for obtaining oil from the water of distillation; in such case only the jacket is heated, but no direct steam is admitted into the still.

The stills of various construction are generally provided with a manhole for charging and emptying. But time can be saved by arranging them so that the still-head may be lifted off, and placing the substance to be distilled in wire baskets. By these means the entire charge can be placed in and removed from the still by a small crane in a few minutes.

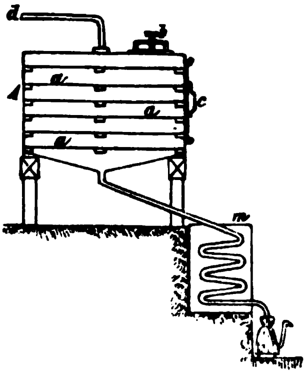
Quite a high pressure of steam can be used for distillation, but, as a rule, it does not exceed four or five atmospheres.

Attempts have been made to effect the distillation of volatile oils without the use of steam by means of hot air, but comparative experiments have shown that less oil is obtained. With the use of steam the vegetable substance swells up by the absorption of water, and thus a free passage is afforded to the oil liberated from the sacs containing it. With the use of hot air, on the other hand, the surface of the plants is completely dried and shrivels to a hard solid mass, which offers considerable resistance to the process of distillation.

This injurious effect of hot air can be somewhat overcome by thorough moistening of the plants to be distilled and allowing the hot air, before entering the still, to pass through a pipe filled with sponges constantly kept wet. But this process offers no advantages over that by steam; the apparatus required is far more complicated, and, besides, a ventilator has to be provided for forcing the hot air through the apparatus.

According to a report in the Journal of the Russian Agricultural Society, 1880, p. 80,* the apparatus (Fig. 275) devised by M. Melnikoff, of Odessa, has been found very efficient in practice. The square box *A* contains a number of metallic perforated diaphragms, upon which are placed, through the door *C*, which can be hermetically closed, the plants to be distilled. Steam is admitted through the pipe *d*, and penetrates the material by a downward current, passing on to a cooler *m*, where it is condensed, and from which the condensed liquid runs into a Florentine flask where the liquid is separated.

FIG. 275.



*Apparatus for distilling in a vacuum.* Schimmel & Co., of Leipzig, in their report, April, 1890, say: "After experimenting for many years, we have finally succeeded in solving the problem of distilling in a vacuum, and in practically applying the process to working on a large scale. The first larger apparatus built in our own shops has now been working several weeks. The results obtained therewith prove without doubt that the distillation of volatile oils under a decreased pressure of air is an acquisition of great importance for our industry. With the magnitude of the vacuum attained the disadvantages due to the use of heat disappear, and as regards quality, the volatile oils produced by the new process will possess evident advantages."

In their later reports the above-mentioned firm does not further refer to this subject, and, as will be readily understood, direct information regarding the apparatus, etc., could not be obtained.

The principal disadvantage of distilling under ordinary atmospheric pressure is that the delicacy of the odor of the volatile oils is readily impaired by the necessary heating, especially when the oils are for some time in contact with the heated metallic parts of the apparatus. Continued contact with hot steam also injures the perfume; in short, in distilling the oils readily acquire the so-called *retort odor* (*goût d'alambic*).

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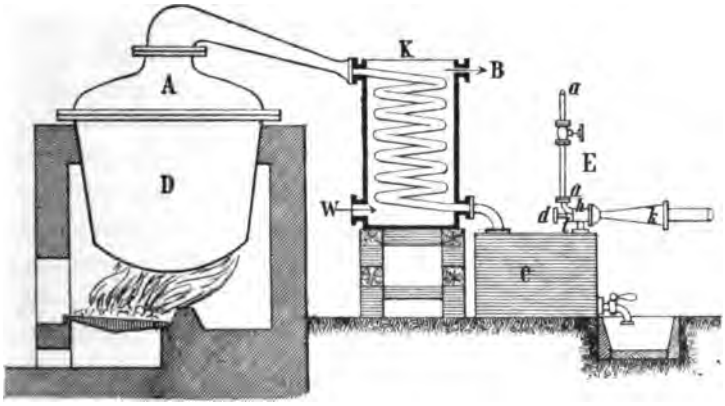
* Dingler's Polytech. Jour., Vol. 223, p. 239.

Evidently this evil can only be remedied by the simultaneous employment of the following means: 1. Distillation at as low a temperature as possible; 2. Rapid conveyance of the vapors from the still; 3. Rapid condensation of the vapors. The first two points can be attained only by distilling in a vacuum, while for rapid condensation a good condenser has to be provided, which with vapors under less than the ordinary atmospheric pressure must, of course, act with far greater energy than with vapors under ordinary pressure.

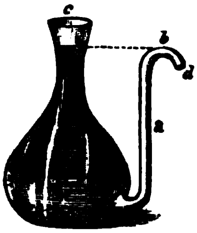
An apparatus for distilling in a vacuum, which admits of

FIG. 276.



many uses and modifications but nevertheless exhibits the arrangement best applied to such still, is manufactured by Körting Bros., of Hanover, and shown in Fig. 276. The retort *D* in the illustration is intended for direct heating, but, of course, can be replaced by any kind of retort. The vapors escape through the head *A* into the condenser *K;* the water for cooling enters at *W*, and passes out at *E*. The products of condensation reach the receiver *C*. On the upper end of this receiver is placed the Körting steam-jet suction apparatus *E*, which continuously sucks the air and vapors from *D* through *K* to *C*, and forces what is not condensed through the pipe *K* into the open air or into the waste steam-pipe. The waste steam or its water of condensation may be conducted into closed coils in the retort *D*, and the rarefaction of the air and the heating of the retort be effected by the same steam.

FIG. 277.



Descriptions of other stills used in various localities for the distillation of oils of lemon, peppermint, sassafras, turpentine, etc., will be found under the heads of the respective oils.

*Separation of the oil and water.*—As previously mentioned, the specific gravity of most volatile oils is less than that of water. This behavior is utilized for the separation of the oil and water by means of a so-called Florentine flask, Fig. 277. It consists in its simplest form of a glass flask provided near the bottom with a pipe *a*, rising vertically to near the mouth *c* of the flask, where it is bent down, as shown in the illustration. The mixed liquid of water and oil drops from the cooling pipe into the flask, and the water *w* being specifically heavier, separates from the oil floating on top, and gradually ascends in the pipe *a*, finally flowing over at *d*. But the oil and water do not remain sufficiently long in the flask to effect a complete separation; after long standing some oil is still separated from the water running off from the flask. To obtain this a second flask larger than the first is placed under the spout *d*, and a third one may be required for oils, the specific gravity of which is but little less than that of water.

For distillation on a large scale it is more advantageous to use the apparatus shown in Fig. 278. It consists of a glass cylinder with conical terminations. It is open on top, and closed on the bottom by a glass faucet. The latter remains closed until the oil almost reaches to the upper edge of the cylinder. The same apparatus can be employed for oils heavier than water. In this case the oil collects on the bottom and the water on top. But as generally far more water distils over than oil, even a large apparatus would be filled in a short time. This is obviated by a narrow lateral pipe near the top of the cylinder, as shown in the illustration, through which the excess of water runs off.

Fig. 278.

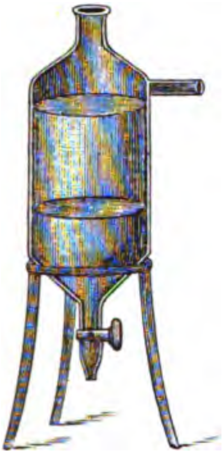
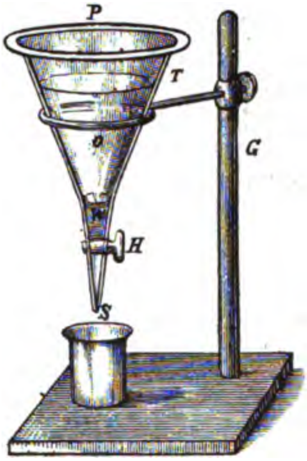


Fig. 279.



The oil collected in the Florentine flask is, however, still mixed with some water, from which it is separated by means of a separator funnel, Fig. 279. This consists of the glass funnel *T* secured to the support *G* and provided with a close-fitting lid *P*.

The fluid is poured into the funnel, the lid placed in position, and the whole allowed to rest until the water *W* is completely separated from the oil *O*. The oil is then separated from the last drops of water by carefully opening the faucet *H*.

### 3. *Process of Extraction.*

The volatile oils, as previously mentioned, dissolve in various fluids, and very readily so in ether, chloroform, carbon disulphide, and petroleum ether. The fluids named have boiling points far below that of water, and are, therefore, especially adapted for the extraction of volatile oil. The process is briefly as follows: A solution of the volatile oil is prepared by suitable treatment of the vegetable substances with one of the mentioned solvents, and the latter removed by distillation, the oil remaining in the still. In practice some difficulties are, however, connected with this process, as, besides the volatile oil, resins and coloring and extractive substances are dissolved, which have to be removed, and also the last traces of the solvent, as otherwise the oil would acquire a foreign odor. Further, the solvents mentioned are very volatile and inflammable, requiring the greatest precautions as regards fire. No lamp or candle should be allowed in the room where the apparatus is located, as even with the most careful construction of the vessels some of the inflammable vapors will escape and ignite.

The extractive agent to be used should, of course, not affect in any manner the odor of the volatile oil, must possess the necessary solvent power for volatile oil and, if possible, none for fat oil, resin, etc. It must further be sufficiently volatile and cheap.

The appended table shows the properties, etc., of extractive agents which may be used:

TABLE SHOWING THE PROPERTIES OF SOLVENTS USED IN THE EXTRACTION OF VOLATILE OIL.[1]

| Names of solvents. | Chemical formula. | Odor. | Specific gravity. | Boiling point, ° F. |
|---|---|---|---|---|
| Carbon disulphide | $CS_2$ | Peculiarly ethereal | 1.292 at 32° F. | 114.8° F. |
| Alcohol | $C_2H_5OH$ | Vinous | 0.80625 at 32° F.<br>0.79367 at 59° F. | 173° F. |
| Ether | $(C_2H_5)_2O$ | Strong, ethereal | 0.736 at 32° F.<br>0.702 at 60.4° F. | 94.8° F. |
| Methyl chloride | $CH_3Cl$ | Ethereal | 0.9915 at —13° F. | —9.4° F. |
| Chloroform | $CHCl_3$ | Sweetish ethereal | 1.5266 at 32° F. | 141.8° F. |
| Acetone | $CH_3COCH_3$ | Refreshing ethereal | 0.8144 at 32° F. | 133.3° F. |
| Petroleum ether | $C_6H_{14} +_n C_{17}H_{16} + C_8H_{18}$ | Strong and peculiarly ethereal | 0.667–0.707 | 149°–158° F. |
| Canadol | Like petroleum ether |  | 0.65–0.7 at 53.6° F. | 140° F. |
| Benzine | $C_7H_{16} +_n C_8H_{18}$ |  | 0.707–0.722 | 176°–212° F. |
| Benzol | $C_6H_6$ | Characteristically aromatic | 0.885 at 59° F. | 177° F. |

For extraction on a small scale the apparatus, Fig. 280, is a very suitable one. It is especially adapted for manufacturers of perfumery and liqueurs, who wish to extract fresh, odoriferous flowers. It consists of a cylindrical vessel *C*, of tin plate, provided on the bottom with the stop-cock *a* and the pipe *b*. The lid *D* fits into a gutter *R*, running around the edge of *C*, and is hermetically closed by water in *R*. The cylinder is filled with the vegetable substance to be extracted, and sufficient petroleum-ether or carbon disulphide to cover it poured in. The lid is then adjusted, the gutter *R* filled with water, and the apparatus allowed to stand quietly for forty minutes. To remove the fluid from the cylinder, the faucet *o* in the lid is first opened and then the stop-cock *a;* the fluid escapes at *b* and is caught in a well-closed vessel. The operation may be repeated once or twice, or the vegetable substance is pressed out by means of a wooden plate and the apparatus filled anew. The faucet *h* serves for emptying the gutter *R*.

FIG. 280.

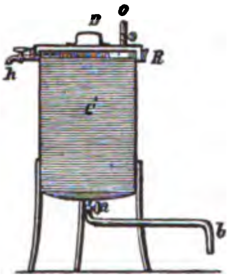
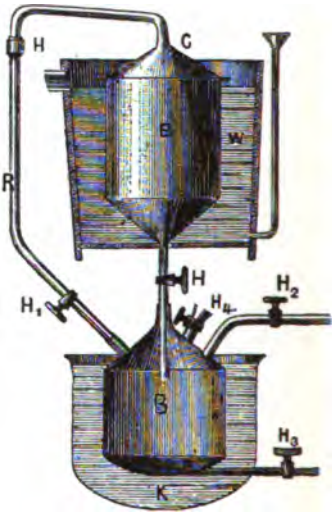


FIG. 281.



The use of this apparatus necessitates the employment of a comparatively large quantity of solvent for the complete solution of the volatile oil, and requires too much time for the extraction, on account of the fluid having to be poured back several times. This disadvantage has been overcome by the construction of an apparatus in which only a certain quantity of solvent is used for the extraction of the vegetable substance, which, as soon as a portion of the oil is dissolved, is at once separated from it and used for the dissolving of fresh quantities of oil. Fig. 281 shows the arrangement of such an apparatus. It consists of two principal parts, the actual extracting vessel *E* and the still *B*. The extracting vessel *E* sits in a vat containing cold water *W*, the arrangement being such that the heated water can be removed and replaced by cold. The still *B* sits in a boiler *K* filled with hot water.

The apparatus is charged in the following manner: The conical head *C* of the extracting vessel *E* is unscrewed, and its connection, at *H*, with the pipe *R*, loosened. The extracting vessel is then charged with the vegetable substance, the head *C* replaced, and the connection with the pipe *R* restored. The cocks $H_2$ and $H_4$ are then opened, and the required quantity of solvent brought into the still. Both cocks are then closed, and the cocks *H* and $H_1$ opened.

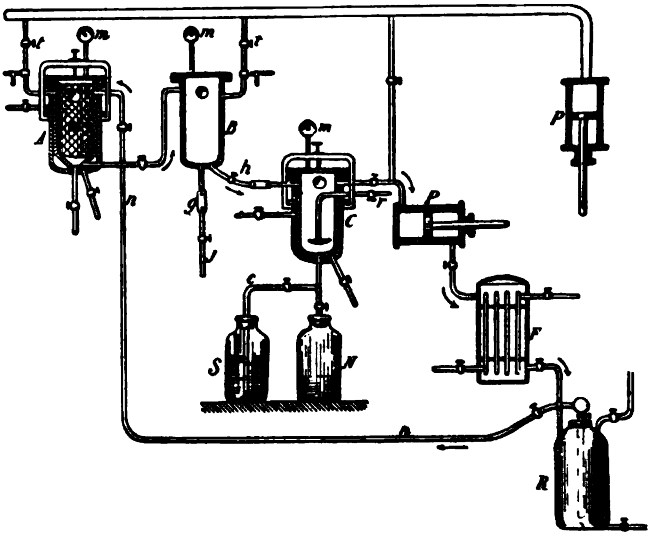
The water in the boiler is then heated until the contents of the still commence to boil. The vapor of the solvent ascends through the pipe *R*; on entering the extracting vessel *E* it is condensed, and, after falling as a spray upon the material to be extracted, finally returns impregnated with volatile oil to the still *B*. Here the solvent is re-vaporized, and passes again through the material in the extracting vessel, while the extracted oil remains in the still. During the boiling of the solvent, the extracting vessel must be suitably cooled by the constant admission of cold water.

When the extraction is finished the cocks *H* and $H_1$ are closed, and the cock $H_2$, which is connected with a cooling-worm, opened. The solvent is then evaporated and regained by condensation. The oil is discharged from the still through a pipe in the bottom provided with the cock $H_3$.

The apparatus can also be so arranged that the still *B* is connected with two extracting vessels which are used alternately; while the contents of one are extracted, the other is emptied and refilled.

Naudain treats flowers or other vegetable substances in a closed apparatus with very volatile fluids, for instance with butyl hydride, or amyl hydride separated from petroleum, or with ethyl chloride or methyl chloride. The vegetable substances in the basket *u* (Fig. 282) are suspended in the receptacle *A*, which is provided with the manometer *m*, and a glass gauge. The lid is then closed, and, after opening the cock *t*, the air is removed by means of the air-pump *p*. The requisite quantity of solvent is then allowed to ascend from the reservoir *R* through the pipe *n*, and in, at the utmost, one-quarter hour is sucked over into the reservoir *B* previously freed from air. The water depositing here is discharged through the pipe *t*, which is provided with the glass *g*. The solution itself is disarged into the distilling apparatus *C*, the cock *h* is closed,

FIG. 282.



and the vapors of the solvent evolved in *C*, at the ordinary temperature of the air, are removed by the pump *P*, and liquefied by strong cooling and corresponding pressure in the cooler *F*. For the maintenance of a uniform temperature a current of air is passed through between the double walls of the distilling apparatus *C*. With a sufficiently low distilling temperature, the fluid discharged into the reservoir *R* is so pure that it can be again used for the extraction of another odoriferous substance. To purify the oil remaining in the reservoir *C* from vegetable wax, etc., alcohol is allowed to ascend from the flask *S* through the pipe *c*. After thorough mixing by the introduction of air through the pipe *r*, the solution is allowed to flow into the flask *N*, cooled down to 14° F. The wax, etc., separating here is filtered from the alcoholic solution at the same low temperature. To regain the solvent remaining in the vegetable substance, the receptacle *A* is heated by the introduction of steam between the double walls and the escaping vapors of the solvent liquefied in a suitable manner.

C. Vincent extracts the volatile oil under increased pressure by means of methyl chloride, which is a colorless gas at the ordinary temperature, but when exposed to a low temperature is a mobile liquid.

Methyl chloride, $CH_3Cl$, is at present prepared on a large scale. It is obtained by the action of hydrochloric acid upon trimethylamine: $N(CH_3)_3 + HCl = N(CH_3)_3HCl$ trimethylamine hydrochlorate; $3N(CH_3)_3HCl = 2N(CH_3)_3 + CH_3NH_2, HCl + 2CH_3Cl$, this reaction taking place at 500° F., and $CH_3NH_2HCl = CH_3Cl + NH_3$, the latter reaction taking place at above 572° F. However, trimethylamine in abundant quantities is obtained in the destructive distillation of the residuary liquors from the manufacture of beet-sugar. To free the technical methyl chloride from admixed basic combinations, it is again treated with hydrochloric acid, dried over calcium chloride, and liquefied under pressure in wrought-iron cylinders. It then forms a mobile colorless liquid of an ethereal odor which boils at —9.4° F., is sparingly soluble in water, but readily in alcohol, and dissolves fats, resins, volatile oils, etc. According to Vincent, it is very suitable for the production of cold, since in boiling it cools the surroundings to —9.4° F. If it is to be used as an extractive agent, it has, however, to be further purified by conducting its vapors through concentrated sulphuric acid and then condensing it.

*Vincent's extracting apparatus* consists of a digester or extractor, which is charged with the plants, a reservoir for liquid methyl chloride, the evaporator or distiller, an air pump and a freezing machine, the former being used for volatilizing the methyl chloride from the solution, and the latter for condensing the vapors of the methyl chloride. The plants are allowed only two minutes in contact with the liquid extractive agent, the solution of perfume being then discharged into the evaporator and the extractor charged with a fresh quantity of methyl chloride. When extraction is finished the higher pressure which prevailed in the extractor during the operation is removed by evacuation; the vapors of methyl chloride thereby sucked on being conveyed by the pump into the freezing machine. Finally the residues have to be steamed, the vapors thereby escaping passing into a gasometer where the steam condenses, while the methyl chloride is conveyed by a pump to a drying apparatus and finally to the freezing machine. The distiller containing the solution of the oil in methyl chloride is heated from the outside by warm water to 86° F., while the air-pump continuously sucks off the vapors of methyl chloride. While at first the pressure in the distiller amounts to 3 or 4 atmospheres, at the end of the operation the manometer indicates a vacuum of ½ atmosphere. The distiller now contains only the volatile oil besides dissolved wax, fat, etc. As a rule, an alcoholic extract of this residue is prepared, since alcohol does not dissolve the above-mentioned foreign extractive substances. Vincent's method can be used not only for substances from which volatile oil is, as a rule, extracted by distillation, but also for obtaining the perfume of plants (jasmine, violets, etc.,) generally subjected to the process of absorption or *enfleurage.*

*Rectification of the oil obtained by extraction.*—The oil obtained by extraction is generally mixed with other substances, consisting chiefly of resin, tannin, and coloring matter. To remove these substances the oil is subjected to purification by distillation. Volatile oil obtained by other methods requires also rectification in order to obtain it entirely pure, as it generally has a yellow color, due to foreign substances, and separates resinous bodies by long storing.

By rectification all oils which have not a characteristic color become colorless and acquire a much finer odor.

For working on a small scale no still is required for rectification; a small apparatus placed in a bath of fat oil, or, better, of paraffin, answering the purpose. By inserting a thermometer in the bath the temperature can be so regulated that the oil is exactly heated to its boiling point and uniformly distilled over.

FIG. 283.

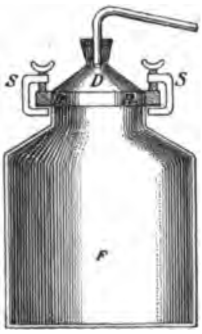
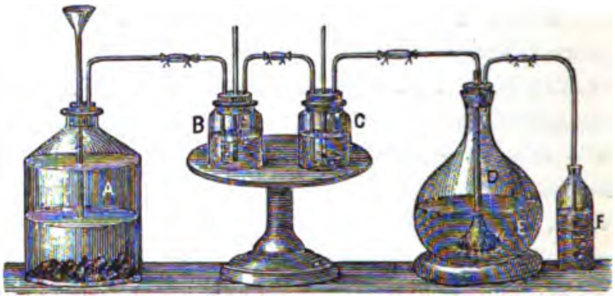


Fig. 283 shows the arrangement of such an apparatus. It consists of a bottle-shaped tin vessel *F*, the conical cover *D* of which is hermetically secured by means of the leather ring *R* and the screw-clamps *S*. A pipe fitted into the cover is connected with a cooling pipe. But the oil prepared by extraction is not sufficiently purified by mere rectification, as traces of the solvent adhere tenaciously to it, which can only be removed by passing a current of air through the oil. But contact with air, as previously mentioned, has an injurious effect upon the delicacy of the odor. For expensive oils a current of air should, therefore, never be used, but one of pure carbonic acid.

Fig. 284 shows an apparatus used for the purpose. The large bottle *A*, filled half full with pieces of white marble, is closed with a cork perforated with two holes; through one of the holes is inserted a funnel tube and through the other a short tube bent at a right angle. The latter is connected with another pipe which reaches to the bottom of the vessel *B*, in which is also inserted a pipe open on the bottom and a short tube bent at a right angle. Alongside *B* stands another vessel *C*, arranged in the same manner. The tube leading from *C* is connected with a tin pipe *D*, with a rose-like expansion on its lower end. This pipe is inserted in the glass balloon containing the volatile oil. Finally, a pipe leads to the flask *F*, filled with water.

To put the apparatus in operation, very dilute hydrochloric acid is poured through the funnel tube upon the pieces of marble, which causes the evolution of a current of carbonic acid. But as the latter carries along water and hydrochloric acid, it has to be freed from them before coming in contact with the volatile oil.

FIG. 284.



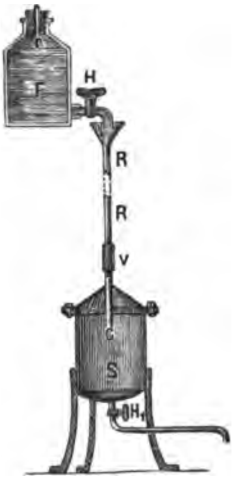
The vessels *B* and *C* serve for this purpose; *B* is half filled with water, while *C* contains strong sulphuric acid. In *B* the hydrochloric acid carried along with the current of carbonic acid is retained, while the water is fixed on the sulphuric acid in *C*. The current of carbonic acid passing out from *C* is perfectly pure and enters the volatile oil through the fine openings in the pipe *D*. It absorbs the traces of solvent still adhering to the oil, and finally passes out through the water in the bottle *F*.

Volatile oil obtained by extraction and purified by a current of carbonic acid will keep for years, without undergoing alteration, if placed immediately in hermetically closed vessels and stored in a dark place. Oil purified by a current of air always becomes somewhat thickly fluid by storing and partly loses its fine odor, which is due to the oxygen absorbed during the process.

### 4. *Process of Extraction with Pressure, or Method of Displacement.*

With an increased pressure the volatile oil may be obtained even at an ordinary temperature by means of one of the previously mentioned solvents, or even strong alcohol.

FIG. 285.



The pressure which a fluid exerts upon the bottom of a vessel, as is well known, depends only on the size of the surface of the bottom and the height of the column of fluid exerting the pressure, but not on the quantity of fluid used. A powerful pressure can, therefore, be exerted upon a surface by means of a very slender but high column of fluid.

By bringing a vegetable substance containing volatile oil together with a solvent under these conditions, a double process takes place: the volatile oil is forced or displaced from the oil-sacs containing it, and at the same time absorbed by the solvent. By the force of the pressure acting upon the vegetable substance the displacement as well as the solution of the volatile oil is very much accelerated and the time required for the work considerably reduced.

Fig. 285 shows the arrangement of the apparatus. The flask *F* is closed by a cork in which is inserted a glass tube drawn out to a fine point; near the bottom the flask is provided with a second neck, with the cock *H* placed over the funnel of a metal pipe *R*. This pipe should be as long as the height of the building will allow; the longer it is the greater the pressure exerted by the fluid will be. A length of thirty-five feet should be the minimum, while the diameter of the pipe need not to exceed one-half to one inch. The pipe is connected with the extracting vessel *C* by means of the coupling *V*. The extracting vessel *C* consists of stout, well-tinned sheet iron, and is provided with the lid *D*, hermetically fitted upon it by means of a leather ring and screws. Immediately over the bottom of the extracting vessel lies the perforated bottom *S*, and a narrow pipe bent laterally and closed by the cock $H_1$ is inserted in the bottom.

All portions of the apparatus must be solid and carefully worked, as the pressure exerted by a column of fluid, even if only thirty-five feet high, is considerable.

The operation is commenced by placing the substance to be extracted in a linen bag fitting exactly in the cylinder *C*. The lid is then adjusted and the fluid in *F* allowed to run into *C* by carefully opening the cock *H*. Care must be taken that the fluid runs in a thin jet down on the inner side of the pipe, so that the air contained in the apparatus can escape without being expelled in bubbles, which would cause a violent ejection of fluid from the pipe. The discharge of the fluid can be readily regulated by suitably widening the mouth of the pipe *R*.

As soon as the fluid rises up to the funnel of the pipe *R*, the cock *H* is closed and the vegetable substance left to the action of the solvent for thirty to sixty minutes. The cock *H* is then very slowly opened and the fluid, which is ejected with great force, is allowed to run into a bottle, in which it clarifies and deposits any vegetable particles.

When all the fluid has run off the cock *H* is closed and the apparatus filled with pure water from a flask arranged in the same manner as *F*. The cock $H_1$ is then opened and the fluid caught in a special flask. This fluid, consisting of water and solution of volatile oil, is, after clarification, separated by a separator funnel. The solution of oil thus obtained is combined with the first, and the oil purified by distilling off the solvent and subsequent treatment with a current of carbonic acid, as previously described.

The method by displacement is suitable for the preparation of oils occurring in considerable quantities in the vegetable substances, as, for instance, oils of cloves, nutmeg, mace, etc.

### 5. *Process of Maceration.*

This process is based upon the affinity of odoriferous substances for fatty bodies, the latter, when impregnated with them, being called *pomades*. These are afterwards made to yield the aroma to strong alcohol, so that finally there is obtained a solution of the volatile oil in alcohol, from which the pure oil is obtained by distilling off the alcohol.

The process of maceration is chiefly used for odoriferous plants yielding volatile oil of such a delicate nature that it would lose considerable of its delicious odor by distillation. The fame of French perfumes is largely due to the fact that all the fine volatile oils used are prepared either by the methods of maceration or absorption.

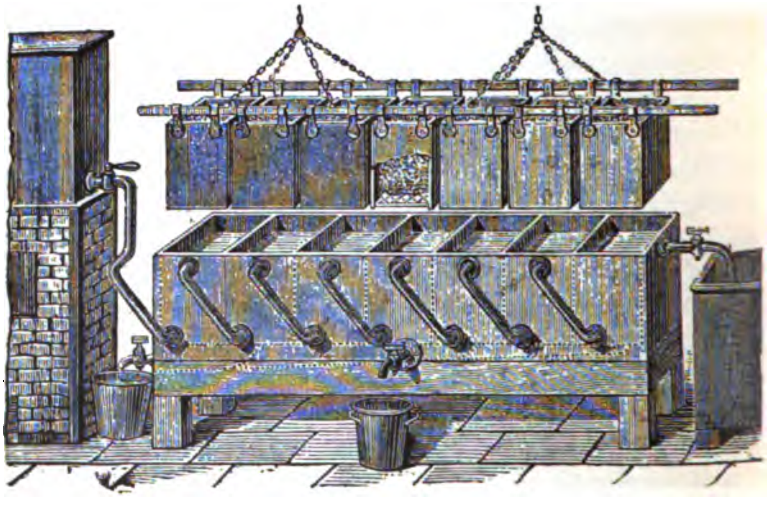
The fat used, olive oil, lard, etc., should be entirely neutral, *i. e.*, free from every trace of acid. The fats are purified by treating them several times in the heat with weak soda lye and then washing carefully with water until the last traces of the lye are removed and the fat shows no alkaline or acid reaction.

With the use of olive oil the so-called "*Huiles antiques*" are obtained, which are merely solutions of volatile oils in the fixed oil. By the use of lard, etc., the genuine pomades are obtained, which are directly used as expensive articles of perfumery, but in the factories serve as the starting-point for the preparation of volatile oils.

The old process of maceration, which is still in use in some parts of France, is as follows: A certain quantity of fat is placed in an enameled iron or porcelain pan provided with a water or steam bath. When the fat is melted the freshly-gathered flowers from which the aroma is to be extracted are thrown in and left to digest for from twelve to twenty-four hours, the fat being kept fluid and stirred frequently. When the flowers are completely exhausted the fat is strained from them into fresh pots, in which it is again macerated with fresh flowers as before. This operation is repeated ten to fifteen times until the pomade has acquired the desired strength. In France these pomades are indicated by numbers ranging from six to twenty-four, according to their strength, the higher number indicating the more costly pomades.

Experience, however, has shown that volatile oil prepared by this process possesses a finer odor the shorter the time the flowers remain in contact with the fat. An apparatus has, therefore, been devised which reduces the time of maceration to the shortest period possible. The kettle to the left (Fig. 286) supplies the fat heated to the proper temperature, which circulates slowly through the macerating tank, in which a constant temperature of 149° F. is maintained by means of a steam-pipe. The macerating tank is divided into compartments, in which baskets containing the vegetable substance to be extracted are suspended. The basket on the left contains the substance which has passed through all the compartments; it is removed from time to time, filled with fresh substance, and

FIG. 286.



then attached to the right, the other baskets being moved to the next compartment to the left. In this way the fresh substance has to traverse each compartment from right to left, while the fat flows slowly from left to right and, saturated with the perfume of the substance, collects in the tank on the extreme right.

The fats impregnated with volatile oil are as soon as possible subjected to further treatment. When olive oil has been used the manipulation to obtain the volatile oil is very simple. The saturated oil is placed in large glass balloons filled about half full with very strong alcohol absolutely free from fusel oil. The balloons, well closed, are placed in a semi-dark and moderately warm room, where the oil remains in contact with the alcohol for several weeks. As the oil forms a layer sharply separated from the alcohol, the contents of the balloon have to be frequently mixed by shaking.

If the manufacturer has at his disposal a cheap mechanical force, the time required for the solution of the volatile oil from the fat oil can be very much reduced by agitating the fat incessantly with the alcohol. The apparatus used for the purpose is very simple, and consists chiefly of a horizonal cylinder revolving slowly around its axle. Through an opening which can be closed fluid-tight by a screw, the cylinder is filled three-quarters full with oil and alcohol and then revolved for a few days. In the course of this time the alcohol has absorbed as much oil as it possibly can, and this alcoholic solution then undergoes further manipulation.

It may be here remarked that it would seem almost impossible to obtain the entire quantity of the dissolved oil by treatment with alcohol, the olive oil and the lard also showing, even after months of continued treatment with fresh quantities of alcohol, the odor of the volatile oil. This, however, cannot be considered a loss; the respective fats may be used for obtaining other volatile oil, or, what is most frequently done, utilized as articles of perfumery.

To give the lard saturated with volatile oil as large a surface as possible it is chopped into small pieces, which are then treated with alcohol. The same purpose may be attained in a more complete manner by a simple mechanical contrivance. This consists of a cylinder closed in front and provided with a discharge-pipe about three-quarters of an inch in diameter. The pomade is brought into the cylinder and pressed out in the form of a thin thread by uniform pressure upon an accurately fitting piston. The fat thread is caught by a circular perforated disk of sheet tin in such a manner that the second layer lies crosswise upon the first. The disks covered with fat are then placed upon brackets in sheet-iron cylinders. When the cylinder is full sufficient alcohol to cover the uppermost disk is poured in, and the apparatus hermetically closed. After about one week one-third of the alcohol is drawn off through a cock placed in the bottom of the cylinder, and replaced by a corresponding quantity of fresh alcohol, the operation being repeated in the course of another week.

The alcoholic solutions of volatile oil obtained by either method contain some coloring matter or resin, but generally in such small quantities that their separation is attempted only in a few cases.

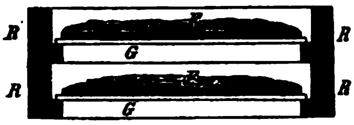
To obtain the oil from the alcoholic extracts, the latter are brought into a distilling apparatus which is placed in a sand or water-bath. Heat is then gradually applied until the contents of the still reach the boiling-point; a higher temperature should be avoided, it being best not to exceed 176° F. At this temperature all the alcohol and the greater portion of the water are evaporated, the volatile oil remaining in the still. The oil is then freed from the last traces of water by means of the separator funnel, which, with very thickly fluid oil, is placed in another funnel filled with hot water. At the higher temperature thus obtained even the volatile oils, which are of a butter-like consistency at an ordinary temperature, will be rendred sufficiently limpid to allow of the complete separation of the water.

### 6. *Process of Absorption.*

This process, called "*enfleurage*" by the French, is chiefly used for procuring the odoriferous principle from such delicate flowers as the acacia, jasmine, tuberose, etc., the delicious odor of which would be greatly modified, if not entirely spoiled, by the application of heat, as in the previously-described processes.

On account of the exceedingly small quantity of volatile oil which the fat absorbs from the flowers by this method, the process is very tedious, requiring much labor and a long time for the saturation of the fat, especially when carried on according to the original French method, which, notwithstanding its faults, is still pursued to a great extent. A series of square glass sheets 2×3 feet is used (Fig. 287), each of which is fitted into a frame with rims three inches in height. Each sheet is then uniformly covered with a thin layer of purified grease, on which the flowers are sprinkled. They are left in this condition undisturbed for from 12 to 72 hours. Some firms use 3000 such frames during the summer season. The frames being filled in the manner described, they are piled one upon the other, the flowers being removed as soon as they have lost their aroma. This is repeated as long as the particular flower continues in bloom, by which time the grease will have acquired a strong scent.

FIG. 287.

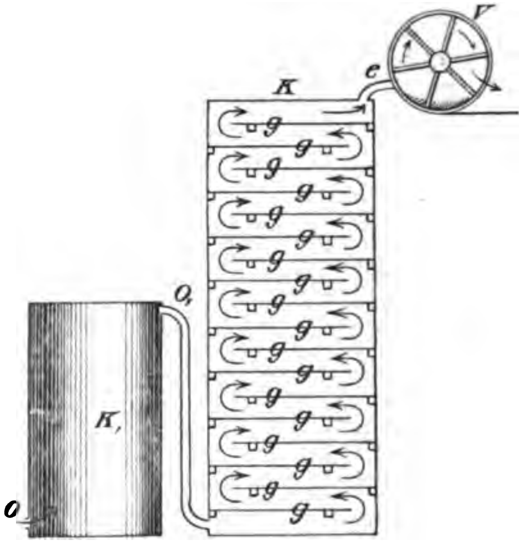


If the odor is to be transferred to an oil, pieces of coarse linen cloth, which have been soaked in the finest olive oil, are laid upon frames which have a bottom of fine linen instead of glass. The flowers are spread on the cloth and left there until fresh ones can be substituted. This operation is repeated for some time, and finally, the oil is pressed out of the linen by a powerful apparatus.

The process of absorption can, however, be conducted with very little expense of labor and time, by the use of an apparatus of simple construction, the principle of which is shown in Fig.

288. Such an apparatus has the further advantage that flowers do not come in direct contact with the fat, whereby a saving of the latter is effected, and it is less liable to rancidity.

FIG. 288.



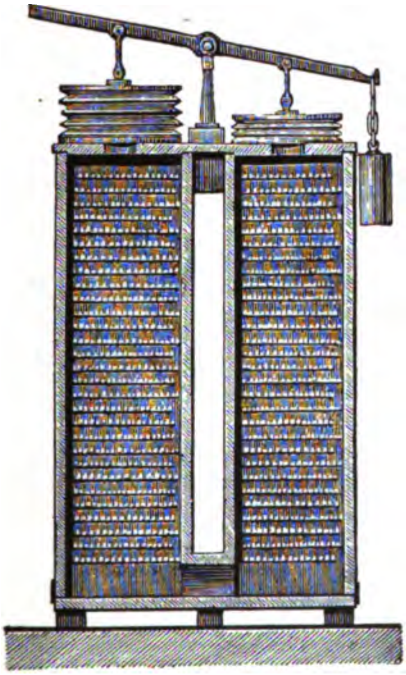
The apparatus consists of a tall wooden box provided with doors, which can be hermetically closed. In the box are placed upon brackets a number of glass plates *g*, arranged in such a manner over each other that, for instance, those with uneven numbers are on the left side, leaving an open space to the right, while those with even numbers are arranged on the right with an open space to the left.

From the bottom of the box a pipe passes into a sheet-iron cylinder $K_1$, filled loosely with flowers, and provided with lateral openings $O$ and $O_1$. From the lid of the box $K$ ascends a pipe *e*, which is connected with a small ventilating apparatus kept in motion by a clock-work and weights.

This ventilator, when in motion, sucks a current of air through the apparatus. The air enters the cylinder $K_1$ at $O$, and, after ascending through the flowers and becoming impregnated with the vapors of the volatile oil, enters through the opening $O_1$ into the box *K*, and, in passing in the direction indicated by arrows, over the plates coated with fat, yields it aroma to them.

Another apparatus for the same purpose, though, in our

FIG. 289.

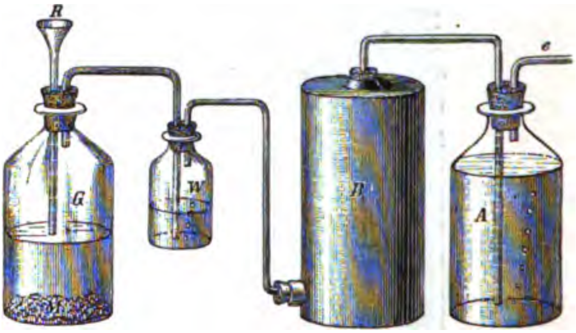


opinion, inferior to the preceding, is shown in Fig. 289. The fat and flowers are placed upon separate trays in an air-tight chamber, arranged as shown in the illustration. The air within the chamber is caused to circulate to and fro by the working of a bellows with which the apparatus is provided, whereby the fat is caused to absorb the odor of the flowers very rapidly, and is less liable to rancidity.

The injurious effect of atmospheric air, previously described, shows itself also in volatile oils obtained by the process of absorption. The yield is smaller, a portion of the oil being rendered odorless by the oxidizing effect of the air. It is therefore recommended to use, instead of air, an indifferent gas, carbonic acid being very suitable on account of its being readily procurable.

Fig. 290 shows an apparatus by which absorption can be effected by means of a current of carbonic acid.

FIG. 290.



The carbonic acid is generated in the flask *F* by pouring hydrochloric acid through the funnel tube *R* upon pieces of white marble *M*. The gas evolved is freed from adhering hydrochloric acid in the wash-bottle *W*, and then passes into the sheet-iron cylinder *B*, filled with flowers, where it becomes impregnated with the volatile oil. It then reaches the flask *A*, containing very strong alcohol, which retains the volatile oil. The carbonic acid escaping through *e* can be again used for the absorption of fresh quantities of oil.

The apparatus described represents the principle of others. For working on a large scale, for instance, the carbonic acid required can be prepared much cheaper by burning charcoal in a specially constructed oven and collecting the carbonic acid in a reservoir, from which it is forced through the boxes filled with flowers and finally re-collected in a second reservoir.

### *7. Process of Absorption with the Assistance of Hot Air.*

As previously mentioned, the principal difficulty encountered in the extraction of volatile oil by means of hot air is the rapid drying out of the external layers of the vegetable substance, thus forming an obstacle to the evaporation of the volatile oil.

The only way to overcome this is constantly to conduct to the fresh flowers and leaves to be extracted as much water as is lost by evaporation; the vegetable substances wilt quickly in the current of hot air, but their surfaces being kept constantly soft, the volatile oil evaporates rapidly.

The apparatus used is as follows: In a kettle set in a brick furnace lies a metal coil, through which a current of air is forced by means of a small pump. This current of air, heated to from 140° to 158° F. by water boiling in the kettle, enters a sheet-iron vessel containing some large sponges kept constantly moist, and finally passes into a box filled with the flowers and leaves to be extracted.

In passing through the box with moist sponges the current of air becomes saturated with aqueous vapor, but on passing through the vegetable substance only absorbs volatile oil. It will be found that a few minutes after the commencement of the operation the current of air leaving the box containing the flowers shows still a temperature of 104° F., and as at that degree the oil is much more volatile than at an ordinary temperature, the time required for absorption will be considerably reduced.

The current of air imprcgnated with vapors of water and volatile oil is conducted through a flask containing carbon disulphide or petroleum-ether, by which the oil is retained. But as these fluids have a very low boiling point, the flask containing them must be connected with a cooling pipe, so that any vapors escaping will condense and return to the flask.

The chief advantage of the use of hot air in connection with the process of absorption is that the time generally required for the work is very much reduced, the volatile oil being obtained entirely pure, in a few hours.

*Storage of volatile oils.* The proper storage of volatile oils is of the utmost importance, since the quality of the oil is much more impaired by errors committed in this respect than by anything else. It may be laid down as a general rule that the oil should be kept in vessels filled full and hermetically closed, in a dark and cool place, because air, light and heat are its greatest enemies. If kept in bottles badly stoppered and only partially filled and in light, warm rooms, the oil loses its fragrancy and acquires a turpentine-like odor; under circumstances it even resinifies.

Up to a certain limit, the *purification of resinified oil* is possible, but it always causes losses. According to J. Curieux, the resinified oil is agitated with a thin paste of borax, bone-black and water for ¼ hour, and then filtered, whereby the resin combines with the borax to a sort of soap, the latter forming with the bone-black a smeary mass, which adheres to the bottle and does not reach the filter. The oil, in a clear and limpid state, runs rapidly from the filter, and again possesses the properties of fresh oil. Resinified oil may also be purified by rectification with water.

According to Askinson, the best way of hermetically closing bottles filled with volatile oil is to draw rubber caps over the stoppers. To prevent drops of oil from adhering to the mouth of the bottle and resinifying, it is also recommended not to pour the oil from the bottle, but to take it out by means of a pipette. As regards keeping the vessels constantly filled, this can in most cases only be done by replacing the oil withdrawn by a corresponding quantity of water. However, this process is not permissible in all cases, for instance, not with lavender oil, which when brought in contact with water, loses fragrancy.

According to Dragendorff, many volatile oils lose, by storing, the property of being miscible in all proportions with alcohol, they yielding clear mixtures only with a small quantity of alcohol. This no doubt is due to resins formed by oxidation, which are precipitated by the alcohol. According to C. A. McDonald, oils of orange peel, lemon and other readily oxidizable oils are best kept by bringing a thick layer of glycerine upon the bottom of the bottle, then filling up with oil and placing the bottle upside down. The oils, it is claimed, keep for months.* It has, however, to be considered that glycerin absorbs odor.

*The residue* obtained in the preparation of volatile oil frequently forms a valuable by-product, for instance, that from the oils of caraway, fennel, anise, coriander, etc. They are often rich in albumen, fat and other nourishing substances, so that they may be used as feed. To make them fit for keeping and shipping, they have to be dried. With extracting apparatus, this is partially effected in the extractor itself, while for the residue obtained in distillation, special drying contrivances have to be employed. For the latter purposes a horizontal cylinder, the bottom of which is heated by steam, may be used. An Archimedean screw slowly conveys the residue over the heated bottom from one end to the other of the cylinder. The vapors evolved escape through a chimney on the upper portion of the cylinder.

*Yield of volatile oils.* The appended table gives the results obtained in the factory of Schimmel & Co., of Leipsic, and without doubt possesses the advantage of being correct:

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Seifenfabrikant 9, 1889, p. 577.

### YIELD OF VOLATILE OILS.

| 100 kilogrammes yield. | Kilogrammes of volatile oil. | Remarks. |
|---|---|---|
| Ajowan seed.......... | 3.00 | |
| Almonds, bitter ....... | 0.40–0.70 | |
| Angelica root ......... | 0.75–1.00 | |
| Angelica seed......... | 1.15 | |
| Aniseed. ............. | 2.80 | Russian. |
| " ............. | 2 40 | Thuringian. |
| " ............. | 3.00 | Spanish. |
| " ............. | 1.30 | Levantine. |
| Arnica flowers ........ | 0.04 | |
| Arnica root........... | 1.10 | |
| Asafœtida ............ | 3.25 | |
| Balm herb............ | 0.10 | |
| Basil herb ............ | 0.04 | fresh. |
| Bay leaves............ | 2.30–2.60 | |
| Bearberry ............ | 0.01 | from *Uva Ursi.* |
| Betel leaves .......... | 0.55 | |
| Birch tar ............. | 20.00 | from *Betula alba.* |
| Buchu leaves.......... | 2.20 | from *Barosma crenulata.* |
| Calamus root.......... | 2.80 | |
| California bay-laurel ... | 7.60 | from *Oreodaphne Californica.* |
| Caraway seed ......... | 4.00 | cultivated German. |
| " ......... | 5.00 | cultivated East Prussian. |
| " ......... | 6.00–7.00 | wild German. |
| " ......... | 3.00 | wild Russian. |
| Cardamoms .......... | 4.00–6.00 | |
| Carrot seed........... | 1.65 | |
| Cascarilla bark........ | 1.75 | |
| Cassia bark........... | 1.50 | from *Cassia lignea.* |
| Cassia flowers......... | 1.35 | |
| Cedar wood .......... | 3.50 | |
| Celery herb........... | 0.20 | |
| Celery seed ........... | 3.00 | |
| Chamomile, German ... | 0.28 | |
| Cheken leaves ........ | 1.00 | |
| Cinnamon, Ceylon ..... | 0.90–1.25 | |
| Cinnamon, white ...... | 1.00 | from *Canella alba.* |
| Cloves ............... | 17.50–19.00 | |
| Cloves, stalks ......... | 6.00 | |
| Coriander seed ........ | 0.80 | Thuringian. |
| " ........ | 0.68 | Dutch, |
| " ........ | 0.15 | East Indian. |
| Crisp mint herb ....... | 1.00 | |
| Cubebs .... ......... | 12.00–16.00 | |
| Culilawan bark........ | 3.40 | |
| Dill seed ............. | 3.80 | German. |
| " ............. | 2.00 | East Indian from *Anethum Sowa.* |
| Elecampane root ...... | 0.60 | |
| Elemi resin ........... | 17.00 | |
| Eucalyptus leaves...... | 3.00 | dry, from *E. globulus.* |
| Fennel flower ......... | 0.30 | |
| Fennel seed .......... | 5.00–5.60 | Saxonian. |

YIELD OF VOLATILE OILS.—*Continued.*

| 100 kilogrammes yield. | Kilogrammes of volatile oil. | Remarks. |
|---|---|---|
| Fennel seed | 2.20 | East Indian from *Foeniculum Panmorium.* |
| Feverfew | 0.03 | |
| Galangal root | 0.75 | |
| Galbanum | 6.50 | |
| Ginger root | 2.60 | African. |
| " | 2.00 | Bengal. |
| Heracleum seed | 1.00 | from *H. Sphondylium.* |
| Hop flowers | 0.70 | |
| Hyssop herb | 0.40 | |
| Iva herb | 0.40 | |
| Juniper berries | 0.50–0.70 | German. |
| " | 0.10–1.20 | Italian. |
| Lavender flowers | 2.90 | German. |
| Lilac flowers | 0.03 | |
| Linaloe wood | 5.00 | from *Elaphrium graveolens.* |
| Lovage root | 0.60 | |
| Lupulin | 2.25 | |
| Mace | 11.00–16.00 | |
| Marjoram herb | 0.35 | fresh. |
| " | 0.90 | dry. |
| Marsh tea or wild rosemary | 0.35 | |
| Masterwort | 0.80 | |
| Matico leaves | 2.40 | |
| Michelia bark | 0.30 | from *Michelia Nilagirica.* |
| Mugwort herb | 0.04 | |
| Mugwort root | 0.10 | |
| Musk seed | 0.20 | from *Hibiscus Abelmoschus.* |
| Mustard seed | 0.75 | German. |
| " | 0.59 | East Indian. |
| " | 0.50 | Russian, from *Sinapis Juncea.* |
| Myrrh | 2.50–6.50 | |
| Nutmegs | 8.00–10.00 | |
| Olibanum | 6.30 | |
| Opoponax | 6.50 | |
| Origanum herb | 3.50 | from *Origanum creticum.* |
| Orris root | 0.10 | |
| Parsley herb | 0.30 | |
| Parsley seed | 3.00 | |
| Parsnip seed | 2.40 | |
| Patchouli herb | 1.50–4.00 | |
| Peach kernel | 0.80–1.00 | |
| Pepper, black | 2.20 | |
| Peppermint | 0.30 | fresh. |
| " | 1.00–1.25 | dry. |
| Peru balsam | 0.40 | |
| Pimento | 3.50 | |
| Poplar buds | 0.50 | |
| Roman chamomile | 0.70–1.00 | |
| Roman cumin | 3.00 | Mogadore seed. |
| " | 4.20 | Syrian. |

YIELD OF VOLATILE OILS.—*Concluded.*

| 100 kilogrammes yield. | Kilogrammes of volatile oil. | Remarks. |
|---|---|---|
| Roman cumin......... | 2.25 | East Indian. |
| Roses................ | 0.05 | fresh, from *Rosa centifolia.* |
| Rosewood ............ | 0.04 | |
| Rue herb............. | 0.18 | |
| Sage herb ............ | 1.40 | German. |
| Sandal wood.......... | 4.50 | East Indian. |
| Sassafras wood........ | 2.60 | |
| Savin herb............ | 3.75 | |
| Saxifrage root......... | 0.03 | |
| Snake root ........... | 2.80–3.25 | Canadian, from *Asarum Canadense.* |
| " ........... | 2.00 | Virginian, from *Aristolochia Serpentaria.* |
| Star anise ............ | 5.00 | Chinese, from *Illicium anisatum.* |
| " | 1.00 | Japanese, from *Illicium religiosum.* |
| Storax ............... | 1.00 | |
| Sumbul root .......... | 0.30 | from *Ferula Sumbul.* |
| Tansy herb ........... | 0.15 | |
| Turmeric root......... | 5.20 | |
| Tussilago root......... | 0.06 | from *Tussilago Petosites.* |
| Valerian root ......... | 0.95 | |
| " ......... | 1.00 | from *Valeriana celtica.* |
| Vetiver root .......... | 0.20–0.35 | |
| Water-avens .......... | 0.04 | from *Geum urbanum.* |
| Water hemlock......... | 1.30 | |
| Wild ginger .......... | 1.10 | |
| Wild thyme........... | 0.20 | |
| Wormseed, Levant .... | 2.00 | from *Artemisia maritima.* |
| Wormwood herb ...... | 0.30–0.40 | |
| Yarrow or milfoil herb . | 0.80 | |
| Zedoary root.......... | 1.30 | from *Curcuma Zedoariæ.* |

# CHAPTER XXI.

## DESCRIPTION OF VOLATILE OILS AND THEIR SPECIAL PROPERTIES.

THE volatile oils, as previously mentioned, may be divided into three groups, viz., the pure hydrocarbons, oxygenated oils and sulphuretted oils. Chemically, this division is, however, of little value, since we find among bodies, which should be classed according to it in one of the groups, combinations varying very much in a chemical respect, and belonging partly in the group of the alcohols, indifferent bodies, acids, etc.

A classification of the volatile oils according to their chemical composition has, therefore, not been attempted, and it has been preferred to arrange them according to their botanical derivation.

### I. *Volatile Oils of the Coniferae.*

The family of *Coniferae* furnishes a large number of volatile oils, which are partly obtained from their balsam—turpentine—partly from the wood, branches and branch-tops, as well as from the needles. The oils are mostly obtained by distillation with water or steam, and partly by destructive distillation. The oils obtained from the turpentine, commonly called oils of turpentine, are the most important. Regarding the physical and chemical properties of the coniferae oils, it may be said in general, that they are specifically lighter than water, boil at from 320° to 338° F., and possess an odor of turpentine. They consist mainly of terpenes, and most of them even of *actual terpenes*. They incline toward oxidation.

*Thuja oil, oleum Thujae* (*Lebensbaumöl*, G.) is obtained by distillation with steam from the leaves and branches of the *arbor vitae*, also called *white cedar* (*Thuja occidentalis*, L.).

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Yield 1 per cent. In a fresh state the oil is colorless, but soon acquires a yellow to yellowish-green color. It is limpid and possesses a pungent, camphor-like odor, reminding one disagreeably of tansy oil. Its taste is camphor-like. Specific gravity, 0.91 to 0.925. The boiling point is not constant, it rising from 320° to 482° F. The oil is sparingly soluble in water, but readily in alcohol or ether. According to Jahn's investigations, thuja oil contains about 10 per cent. terpene (dextro-terpene?), and 96 per cent. thujol, $C_{10}H_{16}O$, besides small quantities of acetic acid and traces of formic acid. By allowing iodine to act upon the oil, Schweiger obtained a hydrocarbon to which he applied the term *thujon*. This body passed over at 329° to 347° F., possessed an odor of oil of turpentine and an acrid taste. By mixing thuja oil with iodine, it dissolves considerable quantities of the latter, and by gentle warming the liquid becomes heated to such an extent that for some time it continues boiling on its own account, whereby the thujon passes over. If the residue be further heated and treated with potash lye, a small quantity of hydrocarbon is separated while the principal mass passes into solution. From the alkaline solution carvacrol is separated. Of other reactions of the oil may be mentioned: Black coloration by potash-lye and resinification by sulphuric acid, as well as by potassium. By the action of potassium no evolution of hydrogen takes place.

*Juniper oil, oleum juniperi* (*Wachholderbeeröl* G., *essence de genièvre*. F.). Much oil is contained in all parts, but especially in the berries, of *Juniperus communis* L., or the juniper, which can be obtained by distillation. The berries used for the purpose should be fully grown, but slightly green, and bruised before being placed in the still. Pure oil of juniper is colorless, thinly fluid, with a sweetish turpentine-like odor and taste. It has a specific gravity of 0.850 to 0.880 at 59° F., boils at 302° to 359° F., and the larger portion of it congeals on exposure to a low temperature. It is soluble in all proportions in glacial acetic acid: It is frequently adulterated with oil of turpentine, which is, however, readily detected by the lessened density and solubility in alcohol. The chief use of juniper oil is for medicinal purposes, it possessing powerful diuretic properties, and in preparing gin, which owes its characteristic aroma to the oil.

The yield of oil varies according to the condition of the berries, ripe green berries yielding, according to Steer, by direct distillation with water 0.4 per cent. Flückiger gives the yield of volatile oil from German berries as 0.7 per cent. and from Hungarian berries as 1.2 per cent.

According to the report of Schimmel & Co., of April, 1890, the actual juniper odor of the oil is due to a substance boiling at over 356° F., which is probably an acetic acid ester. The portions of the oil boiling at a higher temperature consist of sesquiterpene, which yields a hydrochloride fusing at 244.4° F.

The residue from the distillation of juniper berries is used for obtaining *juniper juice* (*Succus Juniperi*) which is used in pharmacy.

*Juniper wood oil* is, according to Maier, a water-clear, limpid oil of specific gravity 0.87; it has an acid re-action, a strong odor of juniper, and a bitter, sharp, balsamic taste. According to Hagen, 100 lbs, of wood yield about 1 lb. of oil. According to Deite, juniper wood oil is nothing but oil of turpentine rectified over juniper wood.

*Cade oil, oleum cadinum* (*Kadeöl*, G., *huile de cade*, F.) is an empyreumatic, dark brown, tar-like liquid obtained by destructive distillation of the wood of *Juniperus Oxycedus*, L., a variety of juniper indigenous to Southern France. Cade oil has recently become of interest because O. Wallach used it to obtain from it sesquiterpene $C_{15}H_{24}$.

Cade oil is used in veterinary practice.

*Savin oil, oleum sabinae* (*Sadebaumöl*, G., *essence de sabine*, F.) is obtained by distillation from the fresh leaves or tops of the common savin, *Juniperus sabina*. In France, where a considerable quantity of the oil is distilled, the yield is about 1 pound of oil from 300 to 400 pounds of the leaves. The oil is pale yellow, and in odor and other physical properties resembles very much oil of turpentine. Its specific gravity ranges from 0.870 to 0.940 at 59° F.; it boils at from 311° to 320° F., and solidifies at 35.5° F. It is but seldom adulterated, as the presence of oil of turpentine or alcohol is readily detected on account of the high specific gravity of the oil and its free solubility in rectified spirits. It is chiefly used in medicine as an anthelmintic, diaphoretic, and emmenagogue.

According to Dumas, savin oil agrees with oil of turpentine in elementary composition, and hence consists of terpene, $C_{10}H_{16}$. According to Wallach, it contains also smaller quantities of a sesquiterpene, $C_{15}H_{24}$.

*Cedar wood oil, oleum cedræ Virginicæ* (*Cedernholzöl*, G.; *essence de cedre*, F.). This oil is derived from the Virginia, American or red cedar (*Juniperus Virginiana*, L.). The oil is obtained by distillation from the shavings of the wood falling off in the manufacture of lead pencils. It consists of two hydrocarbons, one a volatile liquid and the other a solid crystalline compound containing oxygen. At an ordinary temperature cedar oil is a white crystalline body of the consistency of butter. The actual volatile oil separated by pressing is colorless, very limpid, and solidifies only at a very low temperature, but readily resinifies on exposure to the air. Its specific gravity is 0.9622 at 59° F., it boils at 519.8° F., and solidifies at 6° F. On account of its agreeable odor it is frequently used in perfumery.

Another kind of cedar wood oil is obtained as a by-product in lead-pencil works, the vapors given off by the cedar wood being conducted through condensers where the oil is condensed. It consists, however, only of the lower boiling portion, while the really valuable part—which is the higher boiling fraction—remains in the wood. The oil thus obtained is, however, of considerably poorer quality, since it only contains the more volatile portion of the wood.

An oil brought into commerce under the name of *Virginia cedar oil* is obtained from the leaves of *Juniperus Virginiana*, and not from the wood. It has a penetrating, disagreeable odor, and is, therefore, not suitable for perfumery purposes.

According to J. Ch. Sawer, the highly aromatic essential oil of the Jamaica or Honduras cedar is distilled in Jamaica from the *Cedrella odorata*, L. The oils of *Juniperus Bermudiana*, L., and *J. Barbadensis*, L., are also said to be very aromatic.

*Sequoia oil* is obtained from the needles of *Sequoia gigantea*, Torr. (*Washingtonia californica*, Winsl.), the California mammoth tree. G. Lunge and Th. Steinkauler obtained the oil by distilling with steam the needles of trees grown in Zurich for ornamental purposes, and shaking the distillate with ether. By the first distillation they obtained solid oil and by the last distillation only liquid oil. On account of its great solubility the solid crystalline substance was difficult to purify, which was, however, effected by recrystallizing it from warm glacial acetic acid. Its fusing point was at 221° F. and its boiling point at 554° to 572° F. The substance crystallizes in white, slightly bluish fluorescent laminæ, and possesses an extremely penetrating, agreeable, aromatic odor, somewhat resembling oil of peppermint. The composition of this substance corresponds to the formula $C_{13}H_{10}$. It received the name *sesquoiene*, and is isomeric with fluorene (a hydrocarbon from coal tar). After keeping it for several months sequoiene completely loses its odor, and, therefore, the odor is probably due to another body admixed in traces, very likely a terpene also contained in the products from sequoia needles. This terpene was contained in the red brown oil found in the last distillates. It forms the principal mass of the sequoia oil, possesses an agreeable turpentine-like odor and a burning taste. Specific gravity = 0.8522 at 50° F. The terpene has the formula $C_{10}H_{16}$, and is dextro-rotatory. With chlorine it yields a crystallized hydrochloride. Further, the fraction passing over at 440.6° to 446° F., contains the body $C_{10}H_{20}O_2$, which forms a colorless liquid of a very aromatic, peppermint-like odor and extremely burning taste, and is dextro-rotatory. Finally, between 536° and 554° F. a heavy yellowish oil of an empyreumatic, aromatic odor passes over. The chemical nature of the two last liquids is not yet known.

*Turpentine oil, oleum terebinthinae* (*Turpentinöl*, G., *essence de térébenthine* F.) Oil of turpentine together with resin occurs in special oil and resin ducts of the coniferae, particularly in the various species of pine. When these ducts are opened, a mixture of resin and oil of turpentine—hence a balsam—runs out, which is called turpentine. This balsam is soft, transparent to whitish opaque, sticky and draws threads. On exposure to the air it gradually becomes harder, partly by resinification and partly by the evaporation of the volatile oil. When first running from the tree, turpentine is transparent and almost colorless; on exposure to the air—especially moist air—common turpentines become clouded and whitish to brown, while the finer varieties remain clear.

According to Stohmann, the different varieties of turpentine may be classified as follows:

1. *Common turpentines*—

(*a*) *German turpentine* from *Pinus sylvestris*, L., *protunda* and *nigra*, as well as from *Picea vulgaris*, L. (*Pinus abies*, L.) A semi-liquid, viscous mass of a peculiarly disagreeable taste and odor; perfectly soluble in absolute alcohol. Content of oil of turpentine about 32 per cent.

(*b*) *French turpentine* from *Pinus maritima*, Poir. This turpentine dries with greater ease than the German, becomes hard when mixed with magnesia ($\frac{1}{16}$), and yields about 25 per cent. of oil.

(*c*) *American turpentine* from *Pinus palustris* and *P. taeda*, L., but especially from *Pinus, australis*, Michx. Resembles French turpentine, but yields only 17 per cent. of oil of turpentine.

(*d*) *Russian turpentine* from *Pinus sylvestris*, L., and *P. Ledebourii*, Endl. It is not brought into commerce, but only the oil of turpentine obtained therefrom.

2. *Fine qualities of turpentine*—

(*a*) *Venice turpentine from Larix decidua*, Mill. It is at first milky cloudy, but after some time becomes clear and colorless to brownish. It is viscous, draws threads, has a bitter taste and finer odor than common turpentine, dries very slowly, is readily soluble in absolute alcohol, and yields from 18 to 25 per cent. of volatile oil.

(*b*) *Hungarian turpentine* from *Pinus pumilio*, Haenke, is a clear, pale yellow, thinly-fluid mass of a pungent aromatic odor.

(*c*) *Carpathian turpentine* or *cedro balsam* from *Pinus cembra*, L. A clear, colorless thinly-fluid balsam of a juniper-like odor and aromatic pungent bitter taste.

(*d*) *Strasburg turpentine* from *Abies alba*, Mill., is at first milky turbid, but becomes clear after standing. It is more thinly-fluid than Venice turpentine, yellow to brown-yellow, and dries readily. It has a lemon-like odor and a pungent, bitter taste. It is not completely soluble in alcohol; by stirring in $\frac{1}{16}$ per cent. of magnesia, it becomes hard. It yields 35.5 per cent. of volatile oil.

(*e*) *Canada turpentine* or *Canada balsam*, from *Abies balsamea*, Mill. In a fresh state it is limpid, transparent, colorless to yellowish, of an agreeable, turpentine oil-like odor and a pungent taste. Older balsam is yellowish and thicker, and finally dries completely. It contains 24 per cent of volatile oil, and 59.8 per cent. of resin soluble in alcohol, and 16.2 per cent. of resin insoluble in alcohol.

(*f*) *Chian turpentine.* This turpentine is derived from *Pistacia vera* and *P. Terebinthus*, L., of the *Terebinthaceae* family, and actually does not belong here. It is a clear to turbid substance of a greenish-brown to brownish-green color, the consistency of honey, a turpentine oil-like, and at the same time fennel-like, odor, and an aromatic taste reminding one of mastic. It is completely soluble in ether.

*Manner of obtaining turpentine and oil of turpentine.* In the United States the principal source of turpentine is the *Pinus australis*, Michx. (*P. palustris*, L.) commonly called yellow or Georgia pine, a timber highly valued on account of its strength, elasticity and durability. It is found on a belt of country extending from a little south of the Roanoke River, in North

Carolina, through that State, South Carolina, Georgia, Alabama, and Mississippi, and into Louisiana, generally at a distance of 50 to 150 miles from the coast. In North Carolina, where it is chiefly worked, the belt is about 50 miles wide in the north, and it widens as it extends southwestwardly until its northern border crosses the State line about 10 miles west of the Great Pedee River. Its area within the State is about 15,000 square miles, or about 30 per cent. of the State, and its elevation above the sea-level for the most part is less than 200 feet. The soil of this region is generally sandy, and where there is a clay sub-soil the timber is of better quality. On the richer bottom lands other species, of coarser grain, known as the short-leaf, rosemary, and loblolly pines are more common. This last-named pine (*Pinus tæda*) is generally of a poor quality for timber, its sapwood being very thick.

The mode of extracting the crude turpentine is as follows: During the fall and winter the trees are, what is called by the manufacturers of turpentine "boxed:" excavations are made into the trunk of the trees about 6 to 8 inches above the roots. The shape of these so-called boxes is somewhat peculiar: the lower lip is horizontal, the upper arched; the bottom of the box is about 5 inches below the lower lip and 8 to 10 inches below the upper, the capacity varying between ½ and 1 gallon. In a day or two after the boxes are made, the trees are deprived of their bark to the height of about three feet above the box, and also some of the wood is scraped off, in order to allow the so-called "crude" to exude. This is termed "hacking," the hacks being made in the shape of the letter L, and either closed or open. From this the "crude" begins to flow about the middle of March, runs best during July and August, and begins to slacken again in September and October. After the boxes are filled, the "crude" is dipped out by what are called "turpentine dippers," a peculiarly constructed spoon or ladle, into rudely constructed barrels. The trees require scraping every eight or ten days so as to expose a new surface, the flow of the former hacking being clogged by the congelation of resin; a very slight scrape is all that is necessary to set the "crude" flowing again. The number of boxes in a tree depends upon its size. Generally the trees are hardly fit for use after four or five years, the resin not being worth much and yielding little oil; but in some instances the trees are scraped for such a number of years that ladders are necessary to hack the trees afresh, and therefore the oleo-resin, as it flows downwards in the boxes, becomes somewhat congealed and must be scraped off. When dead the tree is cut for tar or sawn into lumber, but for uses requiring strength trees that have been tapped are less serviceable and are often rejected by engineers.

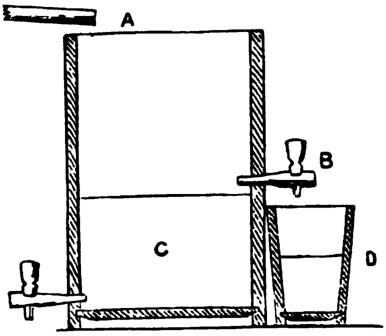
The "crude" is transferred to the stills in the barrels in which it is collected, 10 barrels (weighing 220 pounds each) producing 2 barrels of "spirits" and 6 of "rosin." The flow of the first year is always the best and is therefore called "virgin dip."

The still, which holds between 12 and 20 barrels of "crude," is made of copper in the ordinary shape (the condenser is of the ordinary worm shape), and is set in a brick furnace. It is filled, the dirt, scraps of wood, etc., skimmed off, the head adjusted and luted on. Heat is then applied until the mass attains a uniform temperature of from 212° to 316° F. This is continued until the accidental water, that is, the water contained in the crude as it comes from the forest, has been driven off.

The first product distilled over is pyroligneous acid, formic acid, ether, and methylic alcohol with water. This is known as low wine.

All the accidental water having been distilled off, a small stream of cold water is now let in, so that the heat is kept at or below 316° F., the boiling point of oil of turpentine. The oil of turpentine and water now comes over and the mixture is caught in a wooden tub. This tub, Fig. 291, is constructed as follows: The distillate is caught at *A* from the still and separates into water and oil. At *B* there is an overflow-spout which discharges into the tub *D*. The water is kept low enough in the lower part of the tub to prevent its overflowing through the cock *B* into the receptacle *D*. From this the oil is put into oak casks, well made with iron hoops and securely glued inside.

FIG. 291.



The distiller tests the quality of the flow from time to time in a proof-glass. The distillation is continued until the proportion of fluid coming over is 9 of water to 1 of oil of turpentine. At this stage the heat is withdrawn, the still-cap is taken off, and the hot rosin which remains in a fluid state in the still is drawn off by a valvular cock at the side of the still near the bottom. This rosin passes through a strainer before it reaches the vat, to rid it of foreign substances, such as straw, pine-cones, chips, etc. From the vat it is bailed by wooden buckets fixed on a long handle into the barrels, capable of holding from 280 to 400 pounds. The grade of rosin depends first upon the quality of the turpentine, and second upon the skill in distilling. "Virgin turpentine," if skilfully distilled, will yield "window-glass rosin," of which there are two or three grades. If by any means water gets into prime rosin, it becomes opaque. This accidental addition of water must take place after the rosin has been drawn off from the still.

"Yellow dip" turpentine, which is the running of the second and subsequent years, yields the medium grades of rosin, while the "scrapings," that is, the inspissated gum from the tree-facings, yield an inferior rosin, from very dark to almost black.

Pure American oil of turpentine is colorless, limpid, very mobile, and neutral to test paper. It is soluble in 6 parts of ether and 7½ parts of rectified spirit. It dissolves freely in hot strong alcohol, but on cooling the greater portion re-separates in globules. It is charred by oil of vitriol and strongly attacked by nitric acid, even bursting into flame. It is dextro-rotatory, the rotating power of the rectified oil being, according to Stohmann, $\alpha_D = +14.15°$, the specific rotating power, according to Lafont, $[\alpha]_D = +14° 4'$ and, according to L. Pesci, $[\alpha]_D = + 13.945°$. According to the latter, American oil of turpentine also contains small quantities of levo-rotatory hydrocarbons, possessing, however, but slight rotatory power. Pesci, as well as Lafont, give the boiling point at 312.8° to 314.6° F., while the specific gravity, according to the former, = 0.8641. Wallach found the boiling point of commercial American oil of turpentine = 318.2° to 321.8° F., and that it yields a liquid bromide. From American oil of turpentine a monohydrochloride, $C_{10}H_{16}HCl$, and a dihydrochloride, $C_{10}H_{16},2HCl$, fusing at between 120.2° and 122° F., may be prepared. If the latter is heated with aniline to nearly the boiling point a sudden reaction takes place, aniline hydrochloride being separated and a terpene splitting off. The latter is washed with water and hydrochloric acid and rectified; the greater portion of it passes over at 356° to 365° F., and when bromized yields cinentetrabromide, which fuses at 253.4° to 255.2° F. By heating oil of turpentine to from 482° to 518° F. for a few hours, it may be converted into a hydrocarbon boiling at between 347° and 365° F., possessing an odor like orange peel oil, and, when bromized, yielding large quantities of cinentetrabromide. The oil of turpentine, to be sure, is partly polymerized by heating. In the same manner oil of turpentine is changed when heated with watery or alcoholic nitric or hydrochloric acid, while hot alcoholic sulphuric acid acts differently, the greater portion of the product obtained by heating with the latter passing over at between 356° and 374° F., and yielding a solid tetrabromide $C_{10}H_{16}Br_4$ which forms small lustrous hard prisms, probably of the monosymmetric system.

The principal constituent of American oil of turpentine is dextro-pinene or australene, $C_{10}H_{16}$, besides dipentene. According to Ph. Barbier and J. Hill the australene obtained from

American oil of turpentine is a colorless, very mobile liquid of slight odor and specific gravity = 0.789 at 132° F. By heating at 572° F. for several hours it is converted into isoaustralene which boils at 243.5° F., has a specific gravity of 0.8593 at 32° F. and an odor of oranges. The most usual adulterants of oil of turpentine are certain fractions of petroleum known as "turpentine substitute" and "resin spirit," the most volatile portion of the product obtained by distilling ordinary resin. Certain fractions of shale-oil and coal-tar are not improbable adulterants, but their employment is not common. According to Alfred H. Allen,* the following table gives a number of distinctions between real turpentine and its substitutes:

| | Turpentine oil. | Resin spirit. | Petroleum naphtha. | Shale naphtha. | Coal tar solvent naphtha. |
|---|---|---|---|---|---|
| 1. Optical activity. | Active. | Usually none. | None. | None. | None. |
| 2. Specific gravity. | 0.860 to 0.872. | 0.856 to 0.860. | 0.700 to 0.740. | 0.700 to 0.750. | 0.860 to 0.875. |
| 3. Temperature of distillation, degrees F. | 340 to 356. | Gradual rise. | Gradual rise. | Gradual rise. | Gradual rise. |
| 4. Action in the cold on coal tar pitch. | Readily dissolves pitch to a deep-brown solution. | Readily dissolves pitch to a deep-brown solution. | Very slight action. | Very slight action. | Readily dissolves pitch to a deep-brown solution. |
| 5. Behavior on agitating 3 measures of the cold sample with 1 measure of castor oil. | Homogeneous mixture. | Homogeneous mixture. | Liquid, separates into two layers of nearly equal volumes. | Behaves like petroleum naphtha. | — — |
| 6. Behavior with sulphuric acid. | Almost completely polymerized. | Polymerized. | Very little action. | Considerable action. | Moderate action. |

Although the optical activity of commercial oil of turpentine is variable, it still furnishes a valuable means of distinguishing real turpentine oil from its substitutes and adulterants, all of which are optically inactive.

The specific gravity is alone sufficient to indicate the presence of any considerable portion of the above adulterants of turpentine oil, but it is not of much value for quantitative purposes, owing to the variable character of the shale and petroleum products. In fact, heavier fractions of shale-oil and petroleum are apt to be added to turpentine oil, though their presence is still more objectionable than that of the naphthas.

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* Commercial Organic Analysis, vol. 2.

According to H. E. Armstrong, a good indication of the presence and amount of such adulterants is obtainable by distilling the sample by means of steam. For this purpose a current of open steam, generated by boiling water in a large flask or tin can, is caused to bubble through a definite quantity of the oil contained in a flask or retort fitted to a condenser. Unless it has been freely exposed to the air for some time, genuine turpentine oil leaves a mere trace of volatile matter, but old samples may leave a small proportion (0.5 to 2.0 per cent.) of resinous matter, which solidifies on cooling. As a rule, if more than 0.4 per cent. of non-volatile residue is left after steam distillation, it consists of non-volatilized petroleum oil. This is recognizable by its low density (0.800 to 0.850), and more or less marked blue fluorescence when dissolved in ether. The resin from genuine old oil of turpentine is readily oxidized and dissolved by dilute nitric acid, while the petroleum product is more or less nitrified without altering much in volume. Rosin oil would be recognizable by the formation of a bulky grease on trituration with slaked lime.

The oil which distills over in a current of steam will consist of the genuine oil of turpentine, together with the more volatile portions of any adulterants, and the presence of any shale or petroleum naphtha will be indicated by low specific gravity of the oily portion of the distillate after separation from the water which condensed with it.

B. Redwood has pointed out that the presence of petroleum naphtha in turpentine oil is readily and certainly indicated by the reduced flashing-point of the sample. Thus, while genuine American turpentine oil flashes at 92° F., the addition of only 1 per cent. of ordinary petroleum spirit lowers the flashing-point by about 10° F.

For the detection of petroleum naphtha in oil of turpentine and its isolation therefrom, H. E. Armstrong recommends the following process, which is based upon the different behavior of turpentine and paraffin with sulphuric acid. The paraffins are almost unaffected, whereas turpentine oil is polymerized and for the most part converted into substances of high-boiling points which do not volatilize in a current of steam. Two strengths of acid are employed, one a mixture of 2 volumes of sulphuric acid and 1 volume water (2 : 1 acid), and the other a mixture of 4 volumes acid and 1 volume water (4: 1 acid). The turpentine (500 cubic centimetres is a convenient quantity) is placed with about ¼ to ⅓ of its bulk of 2 : 1 acid in a well-stoppered bottle, and the mixture is somewhat cautiously agitated. It soon becomes more or less heated, and, as it is important to effect polymerization at a temperature not much above the ordinary atmospheric temperature, the bottle is placed in cold water for a short time. After repeated agitation with the acid, the turpentine is converted into a viscid oil, and when this is the case, and no more heat is developed on continued agitation, the contents of the bottle are transferred to a separating funnel, the acid layer is run off, and the oil poured into a flask; the latter having been connected with a condenser and a steam-pot—an ordinary tin can answers admirably—all that is volatile is distilled off. The distillate is mixed with about half its bulk of 4: 1 acid and treated in a precisely similar manner.

The product of this second operation should only consist of a mixture of cymene and paraffinoid hydrocarbon; in bulk it should not be more than 4 to 5 per cent. of the original hydrocarbon ($C_{10}H_{20}$). If much more than about 5 per cent. be obtained, it is desirable to repeat the treatment with 4: 1 acid.

If, from this treatment, it appears probable that petroleum spirit is present, the product is placed in a well-stoppered bottle, together with several times its volume of concentrated sulphuric acid, heated to from 122° to 140° F., with which it is violently agitated. This treatment is repeated if desirable (weak Nordhausen acid being with advantage substituted for the concentrated sulphuric acid), and the residual hydrocarbon is separated, steam distilled, and then measured. The amount thus obtained should not exceed from one-half of one to one per cent. of the original bulk of turpentine oil.

If American petroleum naptha be thus treated, it suffers comparatively little loss, so that the amount of hydrocarbon above one per cent. represents the *minimum* amount of petroleum spirit in the turpentine oil. The naphtha from Scotch petroleum contains a much higher proportion of hydroçarbons alterable by sulphuric acid, and therefore cannot be satisfactorily estimated.

To confirm the presence of petroleum-naphtha the turpentine oil should be distilled. Petroleum-naphtha commences to distill at a temperature which may be above or below that at which turpentine oil boils, according to its quality, but always distills within wide limits of temperature. Turpentine oil commences to boil near 320° F., and almost entirely passes over below 356° F.

The presence of rosin spirit also affects the boiling point in a similar manner. Evidence of the presence of this adulterant is also afforded by the increased yield of hydrocarbons on treatment both with 4: 1 acid and concentrated sulphuric acid, as rosin spirit also yields cymene and paraffinoid by hydrocarbon on treatment with 4 : 1 acid. The cymene from rosin spirit being isomeric with that from turpentine oil, proof of the presence of rosin spirit can be obtained by the detection of its cymene, but this is a somewhat delicate operation.

The chief use of turpentine oil is in the manufacture of varnishes and paints. Rectified oil of turpentine, under the name of "camphene," is sometimes employed for burning in lamps. In medicine it is employed as a vermifuge, stimulant, diaphoretic, etc. Great care must be employed in handling, on account of the extremely penetrating and inflammable nature of its vapor. In stores it is kept in covered tin cisterns.

In the following we give the derivation and characteristics of the various oils of turpentine, according to Dr. Godeffroy, found in the European markets.

*Austrian oil of turpentine*, from *Pinus austriaca* : transparent, colorless or slightly yellowish; specific gravity, 0.864; boiling point, 311° to 314° F.; turns polarized light to the left.

*German oil of turpentine*, from *Pinus sylvestris*, *P. abies*, *P. vulgaris*, *P. picea*, and *P. rotundata*, resembles the former: specific gravity, 0.860 to 0.870; boiling point, 311° to 320° F.; turns polarized light to the left.

*French oil of turpentine*, from *Pinus maritima* : colorless or faint yellowish; specific gravity, 0.860; boiling point, 313° to 315° F.; turns polarized light to the left; odor peculiar; taste burning. French turpentine is chiefly produced in the neighhood of Bordeaux, and yields 25 per cent. of oil.

*Russian oil of turpentine* is obtained from the turpentine of *Pinus sylvestris*, L., and *P. Ledebourii*, Endl., but frequently also as a by-product in the destructive distillation of pine wood. In the latter case it often contains constituents of tar, frequently possesses a penetrating empyreumatic odor, and readily oxidizes. Of all the oils of turpentine mentioned it is of the smallest technical importance. It is dextro-rotatory. It boils, according to Wallach, at 334.4° to 356° F., and in a highly rectified state, according to F. Flowitzky, at 311.9° to 313.5° F. The rectified oil is also free from the empyreumatic odor of the crude oil. Its specific gravity is 0.8764 at 32° F. and 0.86 at 68° F. Its composition has been exactly determined by Wallach. The oil contains as essential constituents dextropinene and sylvestrene, the former passing over at about 320° F. and the latter between 338° and 356° F.; it further contains dipentene, which distils off at above 356° F.

*Swedish oil of turpentine* is prepared in a similar manner to Russian oil of turpentine, resembles it physically and has the same composition.

*Finnish oil of turpentine.* Since the introduction of the preparation of wood tar in closed tar-ovens, the manufacture of turpentine, in Finland, has largely increased. As crude material serve the trunks and roots of *Pinus sylvestris* and *P. abies*. Distillation is effected at a high temperature. The crude oil of turpentine is caught by itself and as much as possible purified from pyroligneous acid, pitch oils and other impurities by direct distillation over lime. Finnish oil of turpentine having thus far not been examined as to its constituents, O. Aschan and E. Hjelt have thoroughly investigated it. For this purpose the oil was subjected to fractional distillation several times repeated, and the separate fractions examined according to Wallach's excellent methods by characterizing the various terpenes by their hydrochlorides, nitroso combinations, etc. Aschan and Hjelt investigated three different kinds of oil of turpentine:

1. *Oil of turpentine from South Finland* obtained by distillation of pine roots in tar-ovens. After fractional distillation repeated five times, the following principal fractions were obtained: 302° to 320° F., 7.1 per cent.; 320° to 329°, 30.2 per cent.; 329° to 338°, 22.6 per cent.; 338° to 347°, 20.1 per cent. A portion of the first fraction was distilled over sodium and well cooled; dry hydrogen chloride was then introduced, whereby a crystalline mass of pinene hydrochloride (fusing point 253.4° to 255.2° F.) was obtained. Pinene nitrosochloride, as well as nitrosopinene, were also prepared and from the former pure pinene, boiling at 310.4° to 312.3° F. was obtained. The portions boiling at a higher temperature were again fractionated, whereby two principal fractions of 338° to 345.2° F. and 345.2° to 352.4° were obtained. These fractions were mixed, each by itself, with ether, and dry hydrogen chloride introduced, under cooling. From the fraction of 338° to 345.2° F. crystals of pure sylvestrene hydrochloride (fusing point 161.6° F.) were deposited, but none from the higher fraction. However, from the latter, as well as from the filtrate of sylvestrene hydrochloride, crystals of a low fusing point were obtained, which after repeated re-crystallization from alcohol showed a fusing point of 120.2° to 122° F., and the composition $C_{15}H_{18}Cl_2$, or that of dipentene dihydrochloride. From the sylvestrene hydrochloride the terpene was regenerated with aniline. The oil showed the characteristic sylvestrene reaction, and boiled at 338° to 341.6° F., while Wallach gives 348.8° F.

for sylvestrene. As regards its constituents, this oil obtained by distillation from wood, therefore, agreed with Swedish and Russian oils.

2. *Oil of turpentine from North Finnland* obtained by distillation from fir trunks, partially as by-product in the preparation of pitch from wood tar. Even in distilling, this oil exhibited different properties from the preceding, the principal mass boiling at a higher temperature. The fractions between 320° and 329° F. were comparatively insignificant, as well as those between 329° and 338°, while those between 338° and 345.2° amounted to 32.2 per cent., and those between 345.2° and 352.4° to 21.5 per cent. In the portions boiling at a lower temperature the presence of much pinene was also established. But no sylvestrene was obtained from the portions boiling at a higher temperature, though it was present in a smaller quantity. The principal constituent was dipentene, a hydrochloride fusing at 122° F. and a tetrabromide fusing at 257° F. being readily obtained. The presence of less sylvestrene and more dipentene is, no doubt, due to the fact that a portion of the oil had been obtained from tar by distillation at a high temperature.

3. *Turpentine*, prepared by distillation with steam from wood rich in rosin. The principal fraction (50 per cent.) boiled at 311° to 320° F., and contained an abundance of pinene. The higher fractions also contained sylvestrene, but no dipentene. This proves that sylvestrene occurs ready-formed in the rosin of the northern *Coniferæ*, while dipentene is not a natural product but is formed by superheating pinene.

In conclusion, it may be mentioned that Aschan and Hjelt oxidized the various fractions of the North Finnish oil of turpentine with nitric acid, and found that terebic acid is chiefly formed from the lower boiling fractions containing pinene, but terephtalic acid preponderatingly from the dipentene fractions.

*Venice oil of turpentine*, from Venice turpentine of *Larix decidua*, is levo-rotatory and resembles the preceding, but has a more agreeable odor. Venice turpentine is mostly obtained in Southern Tyrol and in Piedmont.

Besides these principal varieties the following are also met with:—

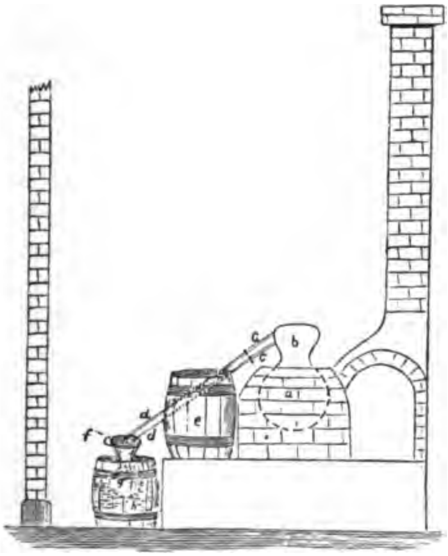
*Dwarf pine oil* (*Krummholz or Latschenoel*), *oleum pini pumilionis*, is obtained by distilling the young tops and cones of *Pinus pumilio* with water. It has a peculiar odor reminding of juniper; specific gravity, 0.865; boiling point, 338° F., and is levo-rotatory.

*Pine-leaf oil* is obtained by distilling the leaves of *Pinus sylvestris* or *P. abies* by means of steam. It has a fine aromatic odor; boiling point, 320° F., and is dextro-rotatory.

*Templin oil* (*Kienoel*) is obtained chiefly in some sections of Switzerland and Tyrol by distilling the wood, branches, leaves, cones, etc., with water. It has a lemon-like odor; specific gravity, 0.860 to 0.880; boiling point, 320° to 327° F., and is levo-rotatory.

In some districts of Thuringia it has been customary for a long time to utilize pine cones for the preparation of oil of turpentine. The cones are gathered from the trees before they

FIG. 292.



are completely ripe. They are then stored in flat sheds, lightly covered with brush, for three to five months, until they have become thoroughly loose. The apparatus used for distilling the oil of turpentine consists of a kettle *a* (Fig. 292) set in brick, surmounted by a tight-fitting copper head *b*, from which two tinned iron tubes *c c*, about the calibre of rifle tubes, project downwards and are joined to similar tubes passing upwards through the condensing barrel *e*, and meeting them. The condensed liquid runs through a funnel *f* into the glass globe *g*, which has an opening on the lower end, closed with a stopper, and floats in the water contained in the barrel *h*. The operations are begun by mashing in a wooden trough with wooden stamps as many pine cones as may be introduced into the kettle, together with one-fifth to one-tenth of their volume of water. The addition of the latter is regulated by the degree of dryness of the cones. In the course of about 12 hours the distillation is completed. The oil obtained has a much finer odor than oil of turpentine. Its specific gravity is 0.868; it boils at 320° to 323.5° F., and is dextro-rotatory.

*General properties of oil of turpentine.* Rectified oil of turpentine is a clear, colorless, very mobile liquid of a peculiar odor, pungent taste and strong refractive power. The odor of the commercial oil is, as a rule, disagreeable because it contains impurities of an empyreumatic nature, as well as products of oxidation. In regard to the refractive power, Riban gives the refractive index of perfectly pure French oil of turpentine as follows: For red, $n_r = 1.4622$;; for yellow, $n_j = 1.4648$; for green, $n_v = 1.4693$; for blue, $n_b = 1.4759$. The specific gravity, as seen from statements previously made, ranges from 0.86 to 0.89. The boiling point lies between 320° and 356° F.; of neutralized oil, rectified in vacuum, according to Bertholet, between 318.2 and 325.4° F., hence close to 320° F. In water the oil is next to insoluble. It gives a clear mixture with eight times its volume of alcohol of specific gravity 0.84, while of alcohol of specific gravity 0.83, 9 to 10 volumes are required. It is miscible in all proportions with absolute alcohol, ether, benzol, chloroform, carbon disulphide, and fat and volatile oils. It is itself a good solvent for sulphur, phosphorus, resins, caoutchouc, etc.

Regarding the *chemical behavior* of oil of turpentine, it may first be mentioned that pure oil of turpentine consists of hydrocarbons of the formula $C_{10}H_{16}$ only, pinene, sylvestrene and dipentene having been determined in it. On these hydrocarbons depends also the behavior of the oil under the action of chemical reagents. It may first be mentioned that oil of turpentine, on exposure to the air, readily absorbs oxygen, and gradually resinifies, carbonic, formic, acetic acids, etc., being formed. The oil thereby becomes more thickly-fluid and yellowish. Naturally, the oil shows a neutral reaction, but when it has suffered a process of resinification it exhibits an acid reaction. The oxidation of oil of turpentine at a low temperature and exposure to the indirect light of the sun yields ozone or hydrogen peroxide. Oil of turpentine is also readily oxidized by other substances, for instance, by fuming nitric acid, it bursting into flame; more slowly by dilute nitric acid whereby acetic, propionic, butyric, paratoluic, terephthalic and terebic acids and dimethylfumaranhydride are formed; chromic acid yields acetic, terebic acids, etc.

*Chlorine* also acts very energetically upon oil of turpentine, withdrawing from it the hydrogen, with which it combines to hydrogen chloride, and separating the carbon; this is generally accompanied by ignition and a discharge of heavy clouds of soot. With a moderate action of the chlorine, products of substitution or addition of the terpene are formed. *Bromine* acts in a similar manner; by sufficiently cooling the oil of turpentine the dibromide $C_{10}H_{16}Br_2$ is formed. *Iodine* dissolves in cold oil of turpentine but yields in a short time, and immediately, when brought into warm oil, an explosive-like reaction. According to R. H. Davies, oil of turpentine as found in commerce takes up 377 per cent. of iodine and hence has the highest iodine number of all volatile oils. If iodine be dissolved in four times its weight of oil of turpentine and the solution subjected to distillation, distillates are obtained up to 482° F., which, according to H. A. Armstrong, contain, besides cymol, a non-aromatic hydrocarbon, $C_{10}H_{20}$ (very likely a mixture of a hydrocarbon boiling at 320° F. and one boiling at 410° F.). The action of hydrogen chloride upon oil of turpentine has already been referred to; by it is formed the artificial camphor hydrochloride, $C_{10}H_{16}HCl$—which forms a white crystalline mass, fuses at 257° F., and boils at 410° F. On the other hand, the dihydrochloride is formed by the action of fuming nitric acid or by conducting hydrogen chloride into an alcoholic or ethereal solution of oil of turpentine; it has the formula $C_{10}H_{16},2HCl$ and forms crystals which fuse at 118.4° F.

When oil of turpentine is exposed to moist air or when it contains water, a hydrate, called terpin, $C_{10}H_{16},2H_2O$, is frequently formed from which several hydrates are derived. The hydrate separating on its own account from oil of turpentine, which is also called turpentine camphor, has the formula $C_{10}H_{16},2H_2O + H_2O$, is a well crystallizing body which by heating loses its water of crystallization, and is converted into terpin.

*The alterations of turpentine oil by heat* seem to commence at 464° to 482° F., and are promoted by the presence of certain substances, such as calcium chloride, sal ammoniac, fluor spar, chloride of zinc, etc. Wallach's investigations have shown the isomeric combination formed to be dipentene.

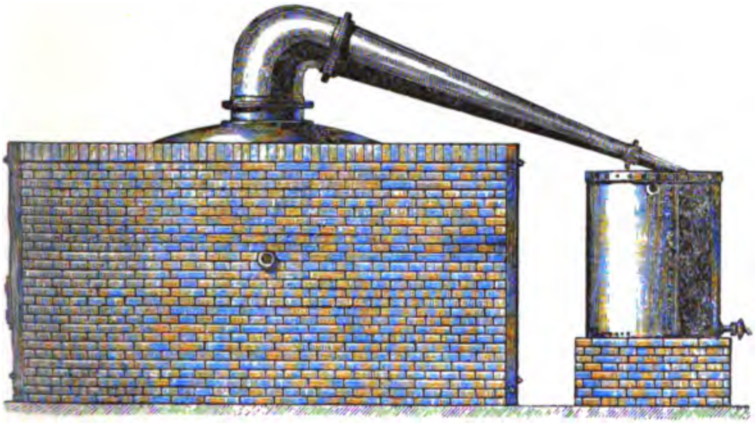
If vapor of oil of turpentine be conducted through a tube not entirely heated to a red heat, there are formed, according to Tilden, isoprene $C_5H_8$, heptine $C_7H_{12}$, toluol, metaxylol, cymol, dipentene and polyterpenes; if the tube is red-hot, there are obtained, according to Schulz, benzol, toluol, meta-axylol and isomeric bodies, further napthalene, phenanthrene, anthracene, etc., hence bodies found in coal tar.

Concentrated sulphuric acid colors good fresh oil of turpentine yellowish-red to dark brown-red, but behaves differently with old varieties of oil; glassy phosphoric acid colors oil of turpentine red.

Closely connected with the production of oil of turpentine is another industry, viz., the distillation of

*Rosin or resin oil.*—The apparatus (Fig. 293) used in the distillation of rosin consists chiefly of an iron still with a low helmet, a cooler, and a receiver. The rosin is introduced into the still, and after closing and luting the charging hole and manhole, heat is slowly applied until the temperature reaches from 316° to 320° F. A light rosin oil—crude pinolin with water—comes over first, which is collected in a glass receiver and then placed in glass balloons until further manipulation. When a cessation in the flow of the distillate indicates that all the pinolin has passed over, the receiver is changed and the heat raised to near the red heat of iron, when the rosin boils, and water and heavy crude oil of rosin distill over together. The crude oil is collected in barrels, while the black residue remaining in the still is used as blacksmith's pitch, or further heated until converted into coke.

FIG. 293.



Pure rosin yields:

| | | |
|---|---|---|
| Pinolin (rosin spirit) | 3.10 | per cent. |
| Rosin oil | 85.10 | " |
| Coke | 3.90 | " |
| Acid water | 2.50 | " |
| Gas and loss | 5.40 | " |
| | 100.00 | " |

The yield from commercial rosin differs of course from the above on account of the presence of pieces of bark, chips, etc., and the contamination of the coke with sand.

The acetic water passing over with the pinolin can be utilized either for the production of acetate of lime, which is sold to the manufacturers of acetic acid, or of acetate of iron, for the use of dyers.

For *acetate of lime* the acetic water is neutralized in large wooden vats, whereby acetate of lime is formed, while a brown insoluble combination of the lime with resinous substances separates on top and is removed. The fluid is filtered and evaporated to 15° B., whereby many resin-like substances are separated. Concentrated solution of Glauber's salt is then added as long as a precipitate of sulphate of lime is formed, but as not all the acetate of lime is converted into sulphate of lime, the portion remaining undecomposed is precipitated as carbonate of lime by means of sodium carbonate. The fluid, freed from the precipitate by filtering, contains now sodium acetate and very little sulphate of lime in solution, and is crystallized by evaporation in shallow cast-iron vessels. The crystals of sodium acetate thus obtained are recrystallized once or twice to free them as much as possible from adhering resinous substances. They are then subjected to fusion, which requires great care and attention, since the degree of temperature at which the salt loses its empyreumatic substances is very close to that at which it undergoes entire decomposition, and the acetic acid is expelled. The salt is first melted in its water of crystallization in a shallow cast-iron boiler, and after evaporation to dryness is remelted at a higher temperature, whereby the mass swells up, evolving oily vapors, at first dark and then gray, and, finally, flows quietly and uniformly. It is then removed from the boiler, and, while hot, dissolved in soft water, and the solution filtered to remove separated particles of carbon. The filtrate is crystallized, then decomposed with sulphuric acid in an iron still lined with lead and provided with a copper or lead helmet, and the acetic acid distilled off. The latter is rectified with some pyrolusite in glass retorts, and should be clear as water without foreign odors.

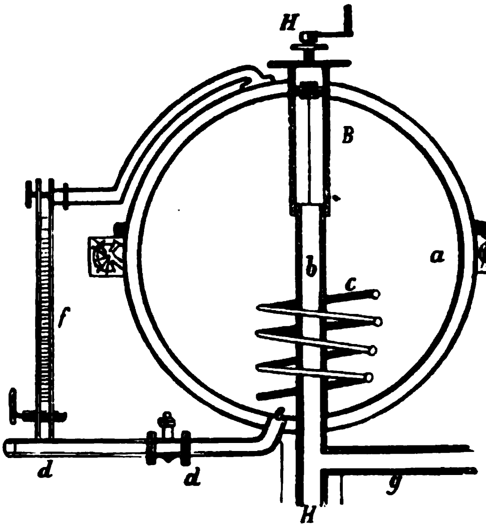
For the preparation of *acetate of iron*, the acetic water is brought into barrels filled with iron filings, waste of sheet iron, etc.

The iron is dissolved in a short time, according to the concentration of the acetic water and the temperature used, so that the saturation of the water is effected in eight to fourteen days, The operation is accelerated by frequently drawing off the liquid, so as to bring the iron in the barrels in contact with the atmospheric air, which accelerates oxidation, and, further, by keeping the acetic water in the barrels at a temperature of from 77° to 88° F., which is best effected by the occasional introduction of steam. Complete saturation is, however, not effected in this manner, and therefore the solution drawn off from the barrels is heated to boiling in cast-iron boilers filled with iron filings. The resinous particles and other impurities collecting on the surface are skimmed off, and the heating continued until the acetic water is completely saturated and the product sufficiently concentrated. It is then evaporated to from 10° to 15° Baumé, and allowed to settle before use. It forms a dark, black-green fluid, and is extensively used in the manufacture of cotton goods.

The light rosin oil or pinolin is occasionally worked into camphene, and for that purpose purified in the following manner: Prepare a milk of lime (1 pound of burnt lime to 10 pounds of water). Add 150 parts of this to 600 parts of crude light rosin oil and stir thoroughly half an hour; then add gradually 15 parts of 90 per cent. pulverized soda and stir again for half an hour. In the course of an hour add 100 parts of soft water. After repeatedly stirring, the fluid is allowed to rest for twelve hours, and then brought into a copper still with good cooling arrangement. The mixture can be distilled either over a moderate direct fire or by means of superheated steam, Fig. 294.

*a* is a spherical vessel surrounded by a steam-tight, wrought-iron jacket; *B* is a hollow column passing through the bottom of the still and projecting into it to about two-thirds of its diameter. The exterior of this column is to be carefully turned so that a second pipe, *b*, can be pushed up and down on it in a manner similar to that of the tubes of a telescope. The movements of this pipe are effected by means of the screw, *H*, fastened in the cover of the man-hole. The effect of this arrangement is that the fluid cannot boil over into the column *B*, and by pushing up and down the pipe *b* the vapors evolved

FIG. 294.



by boiling the contents, which escape through *B* into the cooling apparatus, can be immediately drawn off from the surface of the boiling fluid. The still is further provided with a serpentine pipe, *c*, for the introduction of steam if required. The pipe *d*, which connects with the opening *e*, serves for emptying and charging the still. With this pipe is connected a gauge, *f*, which indicates the quantity of oil in the still. *g* is a continuation of *B*. On the lower end of *B* is a small vessel, *H*, provided with a discharge-cock not shown in the illustration, which catches small quantities of oil accidentally flowing over into *B*. The advantage of this apparatus is in a more complete and better separation of the light from the heavy oil.

The oil passing over first with water is generally slightly yellow, and is preserved for the next distillation. The distillate next obtained, which is clear as water, is shaken with distilled water until all mucus is separated, and then filtered through white blotting-paper into glass balloons or tin cans. The camphene must be stored in a cool place and carefully guarded against access of atmospheric air, as otherwise it easily resiniifies. Pure camphene is clear as water, should not smell too much of oil of turpentine, and should burn with a while flame without depositing soot.

When stored in barrels the crude rosin oil acquires a dark violet color, and is then known as " blue rosin oil." This crude oil is rectified by boiling a whole day with water, the water lost by evaporation being constantly replaced. The next day the water is drawn off, and the rosin oil saponified with caustic soda lye of 37° B. The nearly solid mass is then distilled as long as rosin oil passes over. The product thus obtained is known as rectified rosin oil, and is stored over a thin layer of gypsum in iron tanks, whereby an anhydrous, clear oil is obtained in a few weeks.

By repeating the entire operation, double-distilled or prime rosin oil is obtained. The residues from both operations are melted together with blacksmith's pitch.

In Kraemer and Flammer's apparatus (Fig. 295) the rosin is distilled in a vacuum over an open fire. The quality of the product is much superior to that obtained by the ordinary method of distillation. The vacuum is created by a steam-jet suction apparatus (Fig. 255, Vol. 2, page 221).

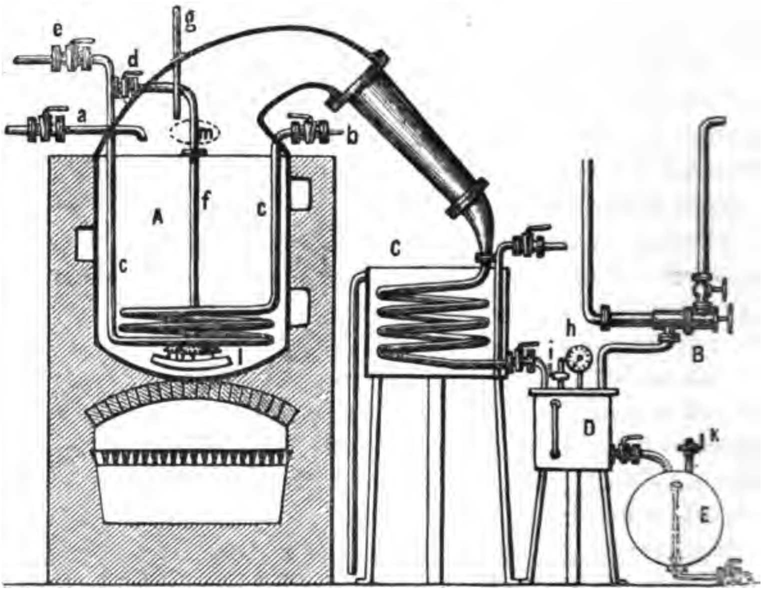
A still, containing 5000 pounds of rosin, yields by distillation with the suction apparatus:—

| | | | |
|---|---|---|---|
| About | 150 | pounds | rosin spirit—pinolin. |
| " | 2500 | " | light-colored rosin oil. |
| " | 850 | " | blue rosin oil. |
| " | 750 | " | green rosin oil. |

The distillates are separated in the order mentioned.

After removing the acid water as carefully as possible, the light-colored rosin oil is purified by treating with caustic soda lye and direct steam until the undecomposed colophony contained in the crnde oil separates a brown soap. The whole is then allowed to rest a few hours, when the lye and rosin soap formed are drawn off, and the oil washed twice with warm water, with simultaneous use of direct steam. The purified, but still turbid oil is then drawn off from the wash water and exposed for some time at a higher temperature to the action of air and light. In this manner the water is entirely separated, and the oil obtained is of a beautiful yellow color, free from rosin and acid, non-fluorescent, almost odorless, and can be advantageously used as machine oil.

FIG. 295.



KRAEMER AND FLAMMER'S DISTILLING APPARATUS.

*A*, still; *B*, vacum apparatus; *C*, cooler; *D*, receiver; *E*, reservoir; *a*, supply pipe; *b*, steam-cock pipe; *c*, worm; *d*, *e*, cocks; *f*, pipe; *g*, thermometer; *h*, vacuum meter; *i*, *k*, cocks; *l*, receptacle for soda; *m*, *n*, steam pipe.

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The blue and green oils are brought into commerce as crude-rosin oils, and are preferably used by the manufacturers of axle-grease. By boiling with slaked lime or granulated zinc, or by treatment with caustic-soda lye, and repeated distillation, the above-mentioned prime rosin oil is obtained.

Commercial rosin oil is a peculiar product. It is not free from oxygen, is thickly-fluid, transparent, yellow to red-yellow, with a decidedly bluish cast, and has an agreeable odor. Its specific gravity is from 0.900 to 0.950 at 59° F.; it boils at 392° F., shows a slight acid reaction, does not resinify on the air, and does not form soaps with alkalies, but only emulsions, as may be shown by the following experiment: Mix 1 cubic centimeter of the heavy oil with 3 cubic centimeters of concentrated caustic soda lye by forcible shaking; then add 5 cubic centimeters of 90 per cent. alcohol, mix and heat to boiling. If the mixture is allowed to rest, the lye separates clear and colorless; the layer of the oil collects over it, and over the oil, the alcohol in a somewhat turbid state.

Only by boiling with slaked lime for some time can the oil be combined to a dark-brown, soft, butyraceous mass, which possesses the ability of absorbing a further quantity of rosin oil. This so-called rosin-oil lime soap is used under the name of *English patent axle-grease.*

The prime or double-distilled rosin oil boils at 455° F., and shows a slighter yellow color, with a scarcely perceptible bluish cast. The latter can be removed by heating with nitrobenzine and dinitro benzine, naphthaline and carbon bisulphide.

The oil has a somewhat bitter and acrid taste, and, when thoroughly purified, does not resinify on exposure to the air. As its consistency does not change at 5° F., it is used as a lubricant. The decolorized oil is also used as an adulterant of vegetable fat lubricating oils, especially of oils of colza and olive. The ordinary rosin oils serve for the sophistication of train oils, and are themselves adulterated with as much as 30 to 40 per cent. of petroleum naphtha and paraffine oil.

Besides, as a lubricant, rosin oil is extensively used for other purposes, for instance, in the manufacture of printing ink, for the production of several kinds of brewer's pitch, the fabrication of transparent bottle lacquers, etc.

In England the crude heavy rosin oil is again distilled with superheated steam, and the distillate mixed 2 hours with 10 per cent. of sulphuric acid. After resting for 20 hours, it is separated from the acid and again distilled by means of superheated steam (Fig. 295), with an addition of milk of lime for the retention and neutralization of the last traces of acid. The condensed vapors pass first through a vertical serpentine pipe, from the lower bends of which the oil, condensed in every curve, is conducted away by means of special small pipes, and thus separated into oils of different degrees of volatility. The end of the serpentine pipe passes in several curves through a vessel with cold water, thus condensing the last and lightest oil vapors. The rosin oil obtained is bleached with chloride of lime, and is chiefly used in the manufacture of white-oil varnishes and for machine oils.

Melsen invented a process for converting heavy rosin oils into light volatile oils. For this purpose the oils, either by themselves or mixed with rosin or other residues of distillation, are allowed to run into a retort heated to a red heat and filled with copper filings or pumice stone, the pores of which are filled with finely divided copper. A current of the pure or mixed rosin oil is then conducted upon the glowing contents and the vapors escaping from the retort condensed. But by this method the formation of many gaseous products which cannot be condensed is unavoidable, and the process can only be used to advantage where the gases can be utilized for illuminating.

Melsen purifies the crude oils by shaking with a solution of sodium carbonate and then distilling with steam. The distillate obtained is treated with 10 per cent. of nitric acid of 30° Baumé, or a mixture of sulphuric acid and pyrolusite, freed from acid with milk of lime, and again distilled with superheated steam.

## II. *Volatile oils of the Monocotyledons.*

The monocotyledons furnish comparatively few and less important volatile oils. Plants of the following families come here into consideration: *Liliaceae, Amyrillidaceae, Iridaceae, Araceae, Gramineae, Zingiberaceae.*

No common properties of the oils derived from these plants can be mentioned, oils from one and the same family frequently exhibiting decided variations. Most oils of the monocotyledons are derived from the bulbs, tubers or roots of the respecttive plants. The oils from the *Liliaceae* contain sulphur, while those from the *Gramineae* contain a camphor $C_{10}H_{18}O$ as an essential constituent. The oils also show considerable variations in their physical properties. Most of the oils here to be considered are specifically lighter than water, though a few are specifically heavier. Some of the oils are liquid, others semisolid, or solid.

### *Liliaceae.*

*Garlic oil, oleum allii sativi* (*Knoblauchöl*, G.; *essence d'ail*, F.) This oil, especially interesting as containing sulphur, is derived from the bulb of *Allium sativum*, or garlic and other species of the same genus. The oil does not pre-exist in the bulb, but is formed in a similar manner as bitter almond oil by a chemical process. The most simple method of production is by submitting the bulb to aqueous distillation. The crude oil is of a dark brownish-yellow color, has an exceedingly pungent, garlic-like odor, and strong, acrid taste, and when applied to the skin produces much irritation and sometimes even blisters; internally it has been used as a vermifuge. It is heavier than water, and boils at 284° F. It may be purified by repeated distillation in a salt-water bath, and is then lighter than water, of a pale yellow color, and not decomposed by boiling. The pure oil combines with nitrate of silver, forming a precipitate soluble in heated alcohol and afterwards separating in crystals. It dissolves with difficulty in water, but readily in alcohol and ether. Allylsulphide $(C_3H_5)_2S$, as shown by Th. Wertheim, forms the principal constituent of garlic oil, and it is not improbable that this combination originally constitutes the entire oil. Of the chemical reactions of garlic oil the following deserve mention: Dilute acids and alkalies produce no effect upon the oil, but nitric acid oxidizes it violently. In concentrated sulphuric acid it dissolves with a purple-red color; when thoroughly dry it is colored indigo-blue by hydrogen chloride, the color disappearing immediately on the addition of water.

*Onion oil*, from *Allium Cepa, L.*, the common onion, is obtained in the same manner as garlic oil. Yield 4.66 per cent. The oil has a red-brown color and a specific gravity of 1.036 at 66.2° F.

### *Amaryllidaceæ.*

*Narcissus oil, oleum narcissæ.*—The narcissus, *Narcissus poëticus*, is cultivated in Southern France for perfumery purposes, but the volatile oil obtained from it by the absorption process does not reach commerce. By experimentally treating the flowers with petroleum-ether a small quantity of oil of a stupefying odor was obtained, which acquired the beautiful narcissus odor only by strong dilution.

*Jonquil oil*, from the flowers of *Narcissus jonquilla*, L., a variety of narcissus indigenous to Southern France. Robiquet has extracted the oil with ether. By evaporating the ethereal extract a yellow butyraceous volatile oil remains behind, which melts in the hand, but boils only at above 212° F. It possesses an agreeable odor and, on cooling, separates yellowish, odorless, sublimable masses of jonquil-camphor. The oil reaches commerce only in the form of jonquil pomade, which is prepared by the absorption process, and possesses an excellent odor.

### *Iridaceæ.*

*Orris root oil, oleum iridis florentinæ* (*Veilchenwurzel*—or *Irisöl*, G.; *essence d'Iris de Florence*, F). The dried rhizomes of *Iris florentina* and *I. Germanica*, natural order *Iridaceæ*, have a pleasant odor, slightly resembling that of violets. It has been used for over 2000 years on account of its fine odor. On jecting the bruised dried root to distillation with water, there is at last found floating upon the condensed water an aromatic substance, solidifying at an ordinary temperature to a crystalline mass, which is highly esteemed by perfumers, and is prepared for commerce by a few large distillers. The price per kilogramme (2.2 pounds) is quoted at 850 marks (about $213) by Schimmel & Co., of Leipzig. The utmost yield of the root, when fully exhausted, is only 0.1 per cent. in solid oil, which is of a light brown color, of the consistency of cerate, and has the delicious odor of violets.

According to Flückiger, the principal mass of the oil is myristic acid, $C_{13}H_{27}COOH$, fusing at 123.8° F., with which is mixed a very small quantity of a thickish, brownish substance, which remains liquid at 12.2° F. This fluid is the bearer of the odor. According to Hager, the oil is solid, pea-yellow, fuses at 100.4° to 104° F., solidifies at 82.4° F., and is soluble in 5 to 6 parts of alcohol.

*Saffron oil, oleum croci* (*Safranöl* G.; *essence de safran,* F.) is obtained by distillation with water or by extraction with ether from the saffron plant, *Crocus sativus,* L. According to Henry, 9 per cent. of oil is obtained. The oil is yellow, thickly-fluid, sinks in water, possesses a saffron odor and pungent, sharp taste, and on exposure to the air gradually becomes solid. In the latter state, it has a waxy appearance and is lighter than water, it having been converted into saffron-stearoptene. Saffron oil boils at 406.4 to 410° F. and is readily soluble in alcohol and ether. It also appears to be soluble in water to a greater extent than other volatile oils, but a decomposition takes place by which the water acquires an acid reaction. The oil is also decomposed by potash lye. It absorbs iodine in considerable quantity. The composition of the oil corresponds, according to Weiss, to the formula $C_{20}H_{14}O_2$.

*Sweet flag oil or calamus oil, oleum calami* (*Kalmusöl,* G.; *Essence d'acore ou de roseau,* F.) This oil, of importance to both the perfumer and the manufacturer of liqueurs, is obtained by distillation from the root of *Acorus Calamus*, or the calamus or sweet flag. It is thickly fluid, of a dark-yellow or red-yellow color, of a very powerful odor and biting taste. It must be protected from light, otherwise it becomes very thick, and, in the presence of air, resinous. Its specific gravity ranges from 0.890 to 0.950; its boiling-point is at 413.5° F. It is readily soluble in alcohol, 1 part of alcohol dissolving 1 part of the oil, and mixes with glacial acetic acid in all proportions. With iodine it forms in a few hours a reddish-yellow viscous mass, evolving thereby yellowish or grayish vapors.

The *Japanese sweet flag* (*Acorus gramineus*, Ait.) also forms at present a raw material for obtaining oil, of which it contains about 5 per cent., while the fresh roots of *Acorus Calamus* yield about 0.3 per cent., and the dry roots 1.4 per cent.

According to J. H. Gladstone sweet flag oil consists almost entirely of a hydrocarbon boiling at 500° F., some *coerulein*—a blue coloring matter—passing over towards the end of distillation. A. Kurbaloff has found two terpenes, $C_{10}H_{16}$, in the oil; one of them boiling at 316.4 to 318.2; it has a specific gravity of 0.8793, dissolves in alcohol and ether, possesses a turpentine-like odor and with hydrogen chloride forms a crystalline combination. The other terpene boils at 482° to 491° F., is of a bluish color, soluble with difficulty in alcohol, but with ease in ether, and does not yield a solid hydrochloride. While the first terpene probably is pinene, nothing definite can be said about the second, especially since it is not pure as indicated by the color.

Sweet flag oil is frequently adulterated, especially with oil of turpentine. This sophistication can be readily detected by the odor in evaporating.

## *Gramineæ.*

*Grass or andropogon oils.* Of the family of grasses (*Gramineæ*) it is especially the variety *Andropogon*, L., which furnishes fragrant volatile oils. This variety, though distributed over the entire globe, is found only exceptionally outside the tropics, and only the tropical plants yield oil. The following species deserve special mention:

1. *Andropogon muricatus*, Retz = *Anatherum muricatum*, Beans, the vetiver plant (Indian: *Khuskhus or Bena*), the rhizome of which is very aromatic and is used as a stimulant as well as an antiseptic. It yields volatile oil.

2. *Andropogon Nardus*, L., the lemon grass, indigenous to Ceylon and Hindostan. All parts of this plant are rich in volatile oil, the so-called *citronella oil.*

3. *Andropogon Schoenanthus*, L., closely resembles the preceding plant, and furnishes the *lemon-grass oil* or *East Indian geranium oil.*

We have thus to distinguish three different grass oils, viz., citronella oil, lemon-grass oil and vetiver oil, the most important of them being

*Citronella oil*, *oleum citronellæ* (*Citronellaöl*, G.; *huile de citronelle*, F.). This oil, as previously mentioned, is derived from *Andropogon Nardus*, L., and is produced almost exclusively in southern Ceylon. The large plantations are provided with regular steam stills, while the poorer class of cultivators use small kettles and primitive distilling contrivances composed of earthen-ware vessels. According to Freudenberg, about 40,500 acres are planted with Andropogon Nardus in Ceylon. A well-conducted plantation produces three crops a year; each good harvest yields 24 bottles of oil (99 lbs. per bottle) from 40½ acres, but in bad years only two crops can be harvested and the yield is only 18 bottles per 40½ acres. The principal difficulty in distilling is the lack of good fire-wood. The production of citronella oil has enormously increased, the annual output being at least 600,000 lbs. The principal markets for the oil are Colombo and Point de Galle.

The oil is limpid, greenish yellow to brown, but the Fisher brand, which is in great demand, is colorless. The odor is agreeable, but comes out in its entire purity only when the oil is much diluted. The specific gravity of the oil ranges, according to Williams, from 0.893 to 0.897 at 60° F.; while, according to Dodge, it is 0.877 at 60.8° F. and 0.875 at 68° F. The oil boils at 415.4° to 426.2° F., or, according to Dodge, at 392° to 464° F. Pure citronella oil dissolves clear, according to Schimmel & Co., in 10 parts of 80 per cent. alcohol, and at 59° F. possesses a specific gravity not below 0.895.

According to F. D. Dodge, citronella oil passes over between 392° and 464° F., leaving behind a thick oily residue. The distillate contains an aldehyde, which forms a colorless liquid of a peculiar odor, is slightly dextro-rotatory, and boils at 395.6° to 404.6° F. Specific gravity of the distillate = 0.859 at 77° F. Citronellic aldehyde, $C_{10}H_{18}O$, is a constituent of the distillate. When the solution of this aldehyde in acetic acid is treated with sodium amalgam, it yields citronellic alcohol, $C_{10}H_{20}O$, a liquid possessing an agreeable rose-like odor, and boiling at 431.6 to 446° F.; specific gravity = 0.8741 at 80° F. This body occurs in small quantity in the oil itself. The aldehyde and alcohol might, according to Dodge, be constituted as follows: $C_4H_9CH = CHCH(CH_3)CH_2CH_2OH$ for the alcohol, and $C_4H_9CH = CHCH(CH_3)CH_2COH$ for the aldehyde. Besides the aldehyde and alcohol, citronella oil contains a terpene, perhaps limonene, boiling at 350.6° F. Hence, the principal constituent of citronella oil is citronellol, $C_{10}H_{18}O$, besides smaller quantities of a terpene (limonene?) and of an alcohol (citronellic alcohol, $C_{10}H_{20}O$).

Regarding other chemical properties of citronella oil, it may be mentioned that, according to Williams, it absorbs 3.35 to 3.43 per cent. of caustic potash, and 186.3 to 191.26 per cent. of iodine. By nitric acid the oil, when heated, is vigorously decomposed whereby a yellow smeary resin remains behind, which dissolves in alcohol with a golden yellow color. According to Gladstone, from citronellol a dibromide is obtained which by heating is resolved into water, hydrogen bromide and cymol.

Citronella oil is adulterated with fat oils and very frequently with petroleum. Schimmel & Co. give the following test:

"One part of citronella oil should give a clear solution with 10 parts of 80 per cent. alcohol. The test can be applied most suitably by means of a graduated measure. The specific gravity of 80 per cent. alcohol is 0.8645. If the sample is adulterated with fixed oil or petroleum, the mixture becomes cloudy and drops of the adulterant are either precipitated or rise to the surface after the mixture has been allowed to stand for 12 hours."

Citronella oil is largely used as a soap perfume, further, as a substitute for verbena oil, and in the adulteration of rose oil.

*Lemon-grass oil*, also called *Indian balm oil*, *Indian verbena oil*, *Indian geranium oil*, *gingergrass oil* or *Indian palmarosa oil*. As previously mentioned, the oil is obtained from *Andropogon Schoenanthus*, L., while according to others lemon-grass oil is produced from *A. citratus*, Indian geranium oil from *A. Schœnanthus* and Turkish geranium oil from *A. pachnodes* (Roscoe). According to Stohmann, lemon-grass oil and Indian verbena oil are identical, as well as ginger-grass oil, Indian palmarosa and Indian geranium oil, while, according to Roscoe, lemon-grass oil and Indian verbena oil, as well as Indian balm oil, are identical on the one hand, and Indian geranium oil and ginger-grass oil on the other. Thus the whole matter is not quite clear. Finally, according to Stenhouse, the oil is derived from *Andropogon Ivarancusae*, which, according to Engler-Prantl, is a variety of *A. Schoenanthus*, but according to others, the plant from which vetiver oil is produced. Schimmel & Co. make the following statement regarding the oils in question: "Lemon-grass is a hard, coarse grass indigenous to the islands west of the East Indies, and requires scarcely any cultivation. The largest quantity of lemon-grass oil is brought from India and the Malay peninsula. The export from Ceylon amounted, in 1885, to about 495 lbs., and, in 1886, to only 333 lbs. During the latter half of 1891 the supply from Cochin amounted to 1196 cases of 12 bottles each = 14352 bottles, weighing about 27,040 lbs. In 1865, the oil cost 1s. 6d. per ounce, and in 1891 it was worth 1½d. per ounce. The ginger grass (*A. Schoen-*

*anthus*) grows wild in large masses in Central India and in the northwest provinces of India. It furnishes, without doubt, the fine geranium or palmarosa oil, as well as the ordinary ginger-grass oil, the latter consisting very likely of special fractions of an unpleasant odor caught separately in distilling the grass. East Indian geranium or ginger-grass oil can seldom be obtained pure, it being, as a rule, adulterated with oil of turpentine." According to Lisboa, there exists still another variety of grass, *Adropogon odoratus*, called "*Gawat wedi*" by the natives, the oil of which is golden yellow, of a mild, sweet odor, and surpasses all other grass oils in quality.

*Lemon-grass oil* has a greenish or brownish color and a balm or lemon-like odor, reminding one also of the odor of the verbena or of pears. Singapore and Ceylon are the principal places of cultivation. Gladstone gives the specific gravity as 0.8741 at 68° F., and the boiling point at 392° F., while, according to Williams, the specific gravity is 0.897 to 0.898, and the boiling point 431.7° F.

*East Indian geranium oil* is chiefly produced in Khandesh (Presidency Bombay). The oil derived from the district Namár or Nimár is called *grass oil of Namár*, while the Turks call it *Idris yaghi* or *Entersha*. The oil is greenish to yellowish brown, and possesses an agreeable aromatic odor, reminding one somewhat of rose oil, but by no means so strong. It has a pungent, but agreeably lemon-like taste.

Lemon-grass oil has been examined by Barbier and Bouveault, who found in it a methylheptenone $C_8H_{14}O$, which combined with sodium bisulphite in a crystalline form, from which the ketone was separated by soda. The ketone boils at 336.2° to 338° F., and has a specific gravity at 32° F. of 0.8632. The authors are of the opinion that the compound is a new methylheptenone, differing from the ketone which Wallach obtained by heating cineolic anhydride and by Tiemann and Semmler through the oxidation of citral, in that it does not give any m-hydroxyol with zinc chloride, nor a solid bromide with bromine and soda solution.

From the synthetical methylheptenone prepared by Barbier and Bouveault by condensation of valeraldehyde with acetone, the new ketone differs in its physical constants and in the products obtained by oxidation with chromic-acid mixture; under these conditions the synthetical body yields valerianic acid, while the natural compound yields acetic acid.

By gentle oxidation of the citral contained in lemon-grass oil, Barbier and Bouveault obtained methylheptenone carbonic acid, $C_8H_{13}OCOOH$. The same process allowed to take place energetically yielded terebinic acid.

The terpene sometimes present in lemon-grass boils, according to Barbier and Bouveault, at 247° F; it takes up four atoms of bromine by direct addition, and yields a tetrabromide melting at 185° F.

In the laboratory of Schimmel & Co. methylheptenone has also been isolated from lemon-grass oil, but was found, contrary to Barbier and Bouveault, to exactly correspond in its properties with the ketone first described by Wallach. The methylheptenone regenerated from the bisulphite compound boiled at 343.4° to 345.2° F. and had a specific gravity of 0.853 at 68° F.; refraction $n_D = 1.43996$ at 68° F. The data given by Wallach for the methylheptenone from cineolic acid are: Boiling point 343.4° to 345.2° F., specific gravity at 68° F. 0.853; refraction $n_D = 1.44003$ at 68° F.

If the ketone of lemon-grass oil be heated with zinc chloride in the manner described by Wallach, an abundant yield is obtained of m-hydroxylol $C_8H_{11}$ (boiling point 269.6° to 275° F.) which is converted on nitrating into dinitro-m-xylol (fusing point 197.6° F.) and further into the very characteristic trinitro-m-kylol (fusing point 357.8° to 359.6° F.) soluble with difficulty in alcohol. From these data the identity of the methylheptenone from lemon-grass oil with the compound of similar composition described by Wallach and by Tiemann and Semmler, cannot be doubted.

From lemon-grass oil an alcoholic body boiling at 388.4 to 392° F. has also been obtained; the investigation of this body is still incomplete. Probably the substance is linalool.

Geraniol, which had been previously discovered in lemon-grass oil, has been prepared in a pure state by means of the calcium chloride compound; it is identical with the geraniol of palmarosa oil, of rose oil, etc.

Lemon-grass oil is adulterated with castor oil as well as with oil of turpentine, and Indian geranium oil generally with the latter but also with alcohol and fixed oils. Both oils are used for perfuming purposes especially for toilet soaps, but not less frequently for adulterating other volatile oils, the Indian geranium oil particularly being employed in the adulteration of rose oil. The oil is especially prepared for this purpose by shaking it with water to which some lemon juice has been added, and then exposed in shallow pans to the sun for 2 or 3 weeks. By this treatment the oil acquires a pale yellow color, loses its sharp odor and smells very rose-like.

*Vetiver or vetivert oil* is obtained from the rhizome of *Andropogon muricatus*, Retz, which is also known as *Cus-Cus* or *Ivaranchusa root;* the latter term, however, applies actually to a variety of *A. Schoenanthus.* The oil is obtained by distillation either from the fresh root in India or from the imported dried root in Europe. The best method is to treat the aromatic water, enriched by repeated distillation with fresh root, with petroleum-ether, and expel the latter by distillation. The yield of oil by this method is about 1 pound from a hundred-weight of the rhizomes. The oil is thickly-fluid, of a red-brown color, and has an intense odor very much like that of oil of orris root. It is much used for scenting toilet articles, as it possesses the valuable property of diffusing a lasting perfume. Its specific gravity is 0.923 at 59° F.; it boils at 515° F.

Nothing is thus far known regarding the chemical composition of vetiver oil. Of chemical reactions the following deserve mention: By boiling with potash lye the oil acquires a red-brown color, while sulphuric acid blackens it, a green resin being separated which is readily soluble in ether but not in alcohol. Nitric acid colors the oil only green at the ordinary temperature, but on heating decomposes it completely, a yellow-brown resin soluble in alcohol remaining behind.

Vetiver oil is frequently adulterated with oil of turpentine, while, on the other hand, it is used itself for adulterating geranium oil. By dissolving vetiver oil in alcohol, a vetiver extract is prepared which is used for various fragrant mixtures, for instance, marechale, bouquet de roi, etc. Vetiver tincture or essence is generally prepared by extracting the root with alcohol.

### *Zingiberaceæ.*

*Galangal oil, oleum galangæ* (*Galgantöl,* G.; *essence de galange,* F.). This oil is derived from the rhizome of *Alpinia officinarum,* Hance, indigenous to China, and more seldom from the rhizome of *A. Galanga,* Willd., growing wild in the Sunda islands. It is obtained by moistening the coarsely ground rhizomes with water and after several hours distilling with steam. The oil is pale greenish yellow, possesses an odor of galangal root and a sharp taste. It is limpid, not very volatile, miscible in every proportion with alcohol and ether, and of specific gravity 0.85. On exposure to the air it thickens and loses its odor. According to Schimmel & Co.'s report of April, 1890, the oil has a specific gravity of 0.921 at 590° F., boils at 338° to 527° F., and contains considerable cineol, the camphor-like odor of the oil being due to the latter. The yield from the root is 0.75 to 1 per cent. of oil. The oil is used in medicine. On account of its high price, it is frequently adulterated with oil of turpentine, copaiva oil and lemon oil.

*Ginger oil, oleum zingiberis* (*Ingweröl,* G.; *essence de gingembre,* F). The rhizome of *Zingiber officinalis* is the raw material for ginger oil; it comes into commerce in two forms—either with the outer integuments present or else removed by scraping. The former kind is called *coated,* the latter kind *uncoated* or *scraped ginger.* According to Schimmel & Co., a company has been formed in China which dries the rhizomes by special machinery and makes them ready for export. From the fresh rhizomes, it is stated, 90 per cent. water and starch is withdrawn, the remaining 10 per cent. forming the valuable aromatic constituent of ginger. By this treatment the rhizome is converted into powder, the aroma of which is far superior to that of other kinds of ginger.

The oil is obtained by moistening coarsely powdered ginger with water, and subjecting it to distillation with steam for at least 28 hours.

Ginger oil is pale yellow to yellow, has a strong odor of ginger, and at first a mild aromatic, and then a somewhat burning bitterish, taste. Specific gravity 0.893; boiling point 474.8° F.

According to J. C. Thresh, the chief mass of ginger oil consists of a sesquiterpene or a mixture of sesquiterpenes, which boils at 473° to 518° F. The oil contains also a dextro-rotatory terpene $C_{10}H_{16}$, and products of its oxidation, as well as cymene; old oils also contain traces of acetic and formic acids.

The terpenes of ginger oil have been examined in the laboratory of Schimmel & Co. Fractional distillation yielded a product boiling between 311° and 329° F. possessing the following properties: Optical rotation + 63° 13′ in a 100 mm. tube; specific gravity 0.864 at 59° F. This fraction yielded no crystallizing nitroso-chloride. It remained liquid in a freezing mixture. Treatment with acetic acid and sulphuric acid produced an acetate which, upon saponification, yielded isoborneol (melting point 413.6° F.) of which the combination-product with bromal melts at 159.8° F. From these data it appears certain that ginger oil contains camphene. In the fraction of ginger oil distilling over at 338° F. phellandrene was found. The nitrite melts at 215.6° F.

Of the reactions of ginger oil may be mentioned: By concentrated sulphuric acid the oil, when slightly heated, becomes red brown, and then dissolves in cold alcohol with a cloudy chocolate-brown color, and in hot alcohol with a rapsberry-red color, whereby a few oily drops appear on the surface. With nitric acid the oil becomes first red, and then violet, whereby it resinifies. With fuming nitric acid it explodes.

Ginger oil is principally adulterated with oil of turpentine and alcohol. It is used in small quantities as an addition to some liqueurs, but at present the alcoholic extract of the rhizome is preferred for this purpose since it also contains the bitter principle.

*Cardamoms oil, oleum cardamomi seminis* (*Kardamomenöl*, G.; *essence de cardamome*, F.). This oil is obtained from cardamoms, the fruits of *Elettaria Cardamomum*, White et Maton, a perennial plant indigenous to Hindostan, more particularly to the mountainous regions of the Malabar coast. The greater portion of these cardamoms go by way of Bombay to London, and are called *Malabar cardamoms.* Another variety is the *Ceylon* or *long cardamoms;* they are obtained from *Elettaria major*, Smith, a plant indigenous to the mountain forests of southern and middle Ceylon. Ceylon cardamoms are of less importance on account of their small percentage of oil, they containing but 3.5 per cent. of it, while Malabar cardamoms contain up to 5 per cent. However, according to the report of Gehe & Co., cardamoms with short round capsules resembling the Malabar product are also brought into commerce from Ceylon. For the purpose of obtaining the oil the cardamoms are comminuted and distilled best with direct steam, the yield of oil being greater than when distilled with water.

The oil is pale yellow and possesses a strong odor and taste of cardamoms, the former being somewhat camphor-like, and the latter pungent. The oil dissolves readily in alcohol, ether, glacial acetic acid and potash lye. Specific gravity 0.92 to 0.9245. Oil from Ceylon cardamoms is, according to E. Weber, somewhat thickish, of a yellowish color, and not entirely clear; it boils at 338° to 428° F.

Cardamoms oil consists chiefly of terpineol, further of dipentene and terpinene, and small quantities of acetic and formic acids, and an unknown crystallizing body. Of the chemical reactions may be mentioned the quite energetic action of iodine, the oxidizing action of nitric acid, which under circumstances may cause ignition, whereby a yellow resin remains behind, and finally the dark red-brown coloration caused by sulphuric acid. Hydrogen chloride does not change the oil.

Oil of cardamoms is frequently adulterated with alcohol as well as with orange-peel oil. It is used in the manufacture of liqueurs and in pastry.

*Grains of paradise oil* (*Paradies-körneröl*, G.,) is obtained from the seed of *Amomum Melegueta*, Rosc., indigenous to Western Africa. According to Husemann, the oil is yellowish, of an agreeable odor, pungent aromatic taste, and specific gravity 0.825. It dissolves in alcohol or carbon disulphide, is levo-rotatory and boils at 455° to 496.4° F. Its composition is said to correspond to the formula $C_{20}H_{32}O$.

According to Engler-Prantl, the seeds of several kinds of *Amomum* are used like cardamoms, only they do not reach the European market; for instance, Siam cardamoms from *Amomum Cardamomum*, L., and bastard cardamoms from *A. xanthioides*, Wall., from Siam and Tenasserim; further, Bengal or Nepal cardamoms from *A. subulatum*, Roxb., from Silkim and Nepal, and Java cardamoms from *A. maximum*, Roxb., from Java.

*Turmeric oil* (*Kurkumaöl*, G.,) is derived from the root of the turmeric (*Curcuma longa*, L.) a perennial plant indigenous to India, and extensively cultivated throughout Southern Asia and in many islands of the Indian Ocean. It is brought into commerce chiefly on account of its yellow coloring matter, but the root also contains a volatile oil which may be obtained by distillation with steam. The oil is limpid, of a lemon-yellow color, penetrating odor and pungent taste, and is lighter than water. It was brought into commerce in 1886 by Messrs. Schimmel & Co. According to Bollegg and Suida, the principal mass of the oil distils over at 428° to 482° F., while above 482° F. boiling and immediate decomposition take place. The principal fraction—the *curcumol*—does not smell much of turmeric, but possesses a strong aromatic odor, has the formula $C_{10}H_{14}O$, and hence is isomeric with carvol, and thymol. According to Flückiger, it differs, however, from carvol, in that it does not combine with sulphuretted hydrogen. The oil also contains phellandrene, $C_{10}H_{16}$.

*Zedoary oil (Zittweröl,* G.) is obtained from the rhizomes of *Curcuma Zedoaria,* Rosc., the zedoary, a plant cultivated in India. The oil is pale yellow, has a camphor-like odor and taste, is turbid, thickly-fluid and heavier than water. It dissolves with ease in alcohol and ether. Regarding its composition, it is only known that it consists of an oil lighter than water and an oil heavier than water.

### III. *Volatile Oils of the Families of Cupuliferae, Myricaceae, Salicaceae, Urticaceae, Piperaceae and Chenopodiaceae.*

The dicotyledonous plants yield by far the greatest number of volatile oils, and hence, to afford a better view of the subject, the oils will be discussed in several sections which, to be sure, have been selected quite arbitrarily. It must, however, be remembered that the object of this arrangement is simply not to divide the subject into too disproportionate sections. In accordance with the botanical classification chosen, the volatile oils derived from the following orders will be here discussed:

II. Sub-division: *Angiospermae.*

1. Sub-order: *Choripetalae and Apetalae.*

Family of the *Cupuliferae, Myricaceae, Salicaceae, Urticaceae, Piperaceae* and *Chenopodiaceae.*

A portion of the volatile oils belonging to this division consists almost exclusively, or entirely, of hydrocarbons, such as the oils of birch tar, poplar bud, hemp, pepper and cubebs. Hop oil, commercial betel oil, and perhaps sweet gale oil, contain chiefly varieties of camphor. The essential constituent of birch bark oil is an ester. It deserves special mention that the oils of hemp, betel and cubebs contain turpenes, chiefly sesquiterpene $C_{15}H_{24}$. As in their chemical compositon, the oils vary also in regard to their physical nature. The oils of birch bark and betel are heavier than water, all other oils lighter. To the actual fragrant oils belong only the oils of birch bark and poplar bud, the other oils being employed on account of their taste or remedial action. Only the oils of birch and cubebs, as well as of hops, are more frequently used.

### *Cupuliferæ.*

*Birch oil, oleum betulæ* (*Birkenöl*, G.; *essence de bouleau*, F.) The bark of the common birch, *Betula alba*, L. is subjected in southern Russia to a sort of destructive distillation, whereby birch tar is obtained. This tar remains liquid at a considerable degree of cold, and is used by the Russians for greasing wagon axles, for rendering roofs water-proof, etc. If this tar be subjected to distillation it yields an oily, brown distillate of a strong odor which has an acid reaction and is lighter than water. By subjecting the distillate to fractional distillation an oil of a yellow color and quite agreeable odor, reminding one at the same time of the odor of birch and of turpentine, passes over at 212° F. This is birch-tar oil, while above 212° F. various dark distillates of an empyreumatic odor are obtained.

The American species of birch, *Betula lenta*, variously called sweet birch, black birch, cherry birch, and mountain mahogany, is remarkable for the aromatic flavor of its bark and leaves, and yields an oil by distillation. In a paper read at the Pharmaceutical Meeting, January 17, 1882, Mr. George W. Kennedy gives an account of the manufacture of the oil as carried on by Mr. A. H. Seidle, of Middleport, Schuylkill county, Pa. In locating a place for the distillation of the oil there are several things to be taken into consideration. First, to have an abundance of material conveniently at hand, so that the supply may not soon be exhausted; and, second, a good supply of water. The species of birch used is the *Betula lenta*, commonly known by the names of cherry-, sweet-, and black-birch. It grows to the height of 75 feet; but as generally found is 10 to 25 feet high. It is found in moist woods, and grows north from New England to Illinois and along the Allegheny regions southward.

The first step in the preparation of the oil is the gathering of the bark, or rather of the tree, as the whole tree is used, except a few small sprouts near the ground. These are left undisturbed, and in five years will be found to have grown to the height of 8 or 10 feet, and are then considered large enough to be cut for the extraction of the oil. It will be observed that from the stumps of trees used this year a new growth may be used five years hence, and so one very five years; this industry, therefore, does not involve the destruction of the trees, as one would naturally suppose. The small trees are preferred, and are gathered by a man, for which he is paid about three dollars delivered, besides the payment to the owners of the land of one dollar per ton for the privilege of cutting the trees, making in all four dollars per ton for the trees delivered at the place where the oil is manufactured.

The trees are then submitted to a chopping machine containing two large knives about 18 inches long and 3 to 4 inches wide, made strong and heavy, which are so arranged that they are operated by a wheel similar to that in the ordinary drug mill; for every revolution of the wheel the knives cut four times, and it requires but a short time to cut a ton of the material into pieces from 1 to 4 inches long, in which condition they are ready for the stills. These consist simply of heavy wooden boxes with copper bottoms, resting on a stone foundation, built about 15 to 18 inches above the surface of the earth, a place being made for fire. Wood is used altogether for fuel. The dimensions of the box or still are 4 x 6 feet and 3 feet high.

The material being ready for distillation, it is placed in the still, as much as this will hold, and a sufficient quantity of water is added to fill the still about one-third full. The still is generally permitted to remain in this condition over night, a fire is made in the morning, and distillation proceeds nicely. The condensers used are of two different kinds, one being the ordinary copper worm placed in a large barrel, while the other is made of copper pipe about two inches in diameter and shaped like the letter U, each wing being twelve and a half feet long, and placed in a trough. A stream of cold water is constantly passing into the barrel and trough, and is carried several hundred feet by a wooden gutter from a clean, clear creek of spring water. The condensed steam in the condenser consists of water and oil. The U-shaped condenser, the distiller says, is more desirable than the other, because the first portion of distillate contains oil, whilst in the old worm style several bucketfuls of water pass out before a drop of oil makes its appearance. As the liquid comes from the condenser a novel contrivance is used for collecting the oil and water, consisting of a Mason's fruit jar (two-quart size), complete. A small tin funnel is placed in the metallic cap of the jar at one side and extends below the cap, the condensed liquid runs through the funnel into the jar, and as the weight of the oil is greater than that of the water, it sinks to the bottom of the receiving vessel. The water runs out through a pipe fastened into the cap on the side opposite to that where the funnel is inserted, is conducted into a bucket, and finally emptied into a barrel, to be used again for the next lot of material. The advantages of using the same water in successive operations are apparent. When distillation ceases the crude oil thus obtained, containing a little water and fragments of vegetable matter, is emptied into a can, with a broad, flat spigot fastened as close as possible to the bottom. The oil is then drawn off through the spigot.

The rectification of the oil is accomplished in a very short time, the process being decidedly simple; but as the perfection of this process has cost Mr. Seidle a considerable sum, this valuable information is withheld.

The odorous principle seems to be thoroughly extracted from the bark, and after the distillation the contents of the still have not the faintest aromatic odor or taste.

It is generally understood, in preparing the volatile oil from birch bark, to be absolutely necessary to submit the material to maceration with water before distillation, in order to develop the oil. The distiller states that he obtains the oil occasionally from birch without previous maceration. Sometimes the bark is covered with ice and snow. As stated before, the material is generally placed in the still the night previous to commence distillation in the morning. The steam generated passes up through the material and extracts the oil in its upward exit, but only about one-third of the material is covered with water in the still. When the bark is macerated the yield is about 20 per cent. larger.

There are three layers of bark on the wood. The outer thin tissue contains no oil; the next or middle layer is of a greenish color and likewise contains no oil; the inner layer next to the wood, which is much thicker than the others and more spongy, yields the oil. Mr. Seidle states that during the month of October, or about the time the leaves fall, little if any oil is made on account of the small yield, hardly reaching 30 per cent. of the yield of any other month during the year. The bark is never dried, but cut up and placed in the still in a green condition and the oil extracted.

Water running from coal mines cannot be used in the distillation, because it contains sulphur and its compounds, which seem to act very destructively on the yield, causing a falling off of at least 75 per cent.; that is to say, instead of obtaining four pounds of oil, but one pound is obtained, and this of a bright blood-red color.

The yield of oil from birch, using the whole tree in a green state, is five pounds from a ton of 2240 pounds, or 0.23 per cent.

The oil obtained as above described is entirely colorless, of a strong and agreeable aromatic odor, and of a sweetish aromatic taste. Its specific gravity is 1.180 at 59° F. It boils briskly at from 424° to 436° F., and the boiling point rises to 442° F., where it remains stationary. The oil on being heated to boiling changes in color from colorless to a light reddish brown. With ferric chloride in water it produces a dark violet color, and with a concentrated solution of soda or potash a solid crystalline mass is formed. Heated with nitric acid orange-yellow crystals are produced, nitrous acid fumes being at the same time given off, and when treated with cold nitric acid crystallization takes place, forming methyl-nitrosalicylate. Treated with muriatic acid, after standing several days, white crystals are formed in quantity.

According to J. H. Gladstone, birch oil consists of an oil with a higher but not constant boiling point and a hydrocarbon related to cymol boiling at 339.8° F. On the other hand, according to Cahours, birch oil contains about 90 per cent. of salicylic acid methyl ester, $C_6H_4(OH)COOOCH_3 = C_8H_8O_3$, and 10 per cent. of gaultherilene, $C_{10}H_{16}$, hence a terpene. According to Pettigrew, the oil contains only the first body and no terpene, which Kennedy confirms in so far as he admits that with defective distilling contrivances the terpene might be "washed out." Trimble and Schröter saponified birch oil with potash lye and then agitated with petroleum ether; 0.447 per cent. of the hydrocarbon gaultherilene passed into solution. Gaultherilene boils at 392° F. and has the composition $C_{15}H_{24}$. From the alkaline solution could further be separated salicylic and benzoic acids and an alcohol boiling at 152.6° to 163.4° F. The same results having been obtained in the investigation of wintergreen oil, the latter may be considered identical with birch oil. Finally F. B. Power asserts that birch oil contains no terpene, no benzoic acid and no ethyl alcohol, and that it is optically inactive.

*Birch tar oil* has a yellow color and a quite agreeable odor. It always possesses an acid reaction and contains a hydrocarbon isomeric with oil of turpentine, besides its products of oxidation. By distillation, first with potash lye and then with lime-water, a pure oil is obtained. This oil is colorless, smells like birch bark, has a specific gravity of 0.847 at 68° F. and boils at 312.8° F. It is readily soluble in alcohol and ether. At 1.4° to —0.4° F. it becomes turbid and deposits a white substance. On exposure to the air it absorbs oxygen, evolves some carbonic acid, turns yellow and gradually resinifies completely. The composition of the pure oil corresponds to that of a terpene, $C_{10}H_{16}$. With hydrogen chloride, of which the oil absorbs about one-half its weight, it becomes black without the separation of a solid hydrochloride.

According to Grossmann, the leaves of the birch also contain 0.36 per cent. of a colorless oil, which possesses a balsamic odor, dissolves in 8 parts of alcohol of 0.85 specific gravity, and solidifies at 14° F.

*Beech tar oil* (*Buchentheeröl*, G). This oil has recently been introduced in the treatment of lung diseases. It is obtained by fractional distillation of beech tar produced by destructive distillation of beech wood from *Fagus sylvatica*, L., the beech. The fraction generally employed is the portion passing over between 176° and 482° F., and having a specific gravity of 0.98. The principal mass passes over between 302° and 482° F., and consists of 30 to 50 per cent. of phenols. The heavy oil of specific gravity 1.053 passes over between 428° and 572° F., and contains about 66 per cent. of phenols.

### *Myricaceæ.*

*Sweet gale*, or *Dutch myrtle oil* (*Galgelöl*, G.), is derived from the leaves of *Myrica Gale*. L., the sweet gale, or Dutch myrtle, a shrub growing in swampy places from the mountains of North Carolina northward to Canada, and in the northern parts of Europe and Asia. The fresh leaves yield 0.65 per cent. of a brownish-yellow volatile oil of an agreeable, balsamic odor and specific gravity 0.867 at 62.6° F. It solidifies at 53.6° F. It is soluble in 40 parts of alcohol of specific gravity 0.875, and is said to contain about 70 per cent. of stearoptene.

### *Salicaceæ.*

*Poplar bud oil*, *oleum gemmæ populi* (*Pappelknospenöl*, G). The buds of *Populus nigra*, L. the black poplar, are furnished with a sticky, resinous coating, and for this reason are used in the preparation of an officinal salve. However, they contain also a volatile oil, which, according to J. Picard, has a specific gravity of 0.9002, boils at 500° to 501.8° F., and has the composition of a diterpene, $C_{20}H_{32}$. The oil is dextro-rotatory; the yield amounts to 0.45 to 0.54 per cent. This oil has been brought into commerce by Schimmel & Co. According to their report the yield is about 0.5 per cent. The oil is pale yellow and possesses an agreeable odor, reminding one somewhat of chamomile. It boils at 491$^{c}$ to 492.8° F., and seems to be a unitary body. It is obtained by distillation from dried buds or points of the poplar.

## *Urticaceae.*

*Hemp oil (Hanföl,* G.) The flowers of *Cannabis sativa,* L, the hemp plant, when scarcely ceased to bloom, yield 0.3 per cent. of pale yellow volatile oil of specific gravity 0.9289. It boils at 492.8° to 496.4° F., is levo-rotatory, and possesses $[\alpha]_D = -10.81°$. According to Personne, the oil consists of liquid cannabene, $C_{18}H_{20}$, and solid cannabene hydrogen. $C_{18}H_{22}$, while L. Volente gives sesquiterpene, $C_{15}H_{24}$, as the only constituent of the oil.

*Hop oil, oleum humuli lupuli* (*Hopfenöl,* G.; *essence de houblon,* F.) The spikes or strobiles of *Humulus Lupus,* L., the hop-plant, contain under the scales golden yellow glands filled with a resinous aromatic bitter substance. These glands or granules may be separated from the flowers by heating or sifting, and are brought into commerce, their principal constituent being what is known as *lupulin.* However, they also contain a volatile oil, lupulin yielding by distillation with water 2 per cent., and hops, by the same treatment, 0.5 to 0.8 per cent.

Hop oil from fresh hops is green, and that from lupulin pale brownish; by rectification with water it becomes colorless. It has a strong, penetrating odor of hops, and a pungent, slightly bitter taste, reminding one somewhat of thyme and origanum. Specific gravity, 0.908 at 60° F.; boiling point, between 257° and 572° F. The oil remains liquid at 1.4° F. It is levo-rotatory, insoluble in water, but imparts its taste and odor to it; it is readily soluble in alcohol. On exposure to the air it turns red.

According to R. v. Wagner, rectified hop oil begins to evaporate below 212° F. Between 257° and 347° F. about ⅙ of the oil distills off; the distillate is colorless and clear, smells slightly of hops and more strongly of rosemary, and probably contains a terpene, $C_{10}H_{16}$, which boils at 347° to 356° F., and does not yield a solid hydrochloride with HCl. At 347° to 437° F. about one-half of the oil passes over; the distillate smelling like crude hop oil. By fractional distillation, a fraction boiling at 410° F. may be isolated, which is readily oxidizable and possesses the composition of a camphor $C_{10}H_{18}O$ (composition, 78.19 per cent. C, 12 per cent. H, 9.81 per cent O, which would correspond to the formula, $C_{10}H_{20}O$, or, still better, $C_{20}H_{38}O_2$.) Finally, at 437° F. to 455° F., a yellowish distillate with only 73.8 per cent. carbon was obtained. The residue amounted to ⅛ of the oil, and was brownish and turpentine-like.

According to Personne, the oil consists of terpene, $C_{10}H_{16}$, and valerol, $C_{12}H_{10}O$, because by oxidizing the oil with nitric acid he obtained therefrom valerianic acid. Kühnemann supposes the existence in hop oil of several hydrocarbons and oxygenated bodies, the quantity of which varies with the age and quality of the hops, and further asserts that oil from sulphured hops yields with acids sulphuretted hydrogen. According to Stohmann, the oil itself shows a neutral reaction, but the water from the distillation an acid reaction on account of a content of valerianic acid.

Of the reactions of the oil the following deserve mention: At the ordinary temperature iodine only colors the oil brown, but on heating acts very energetically and the oil is resinified. Bromine acts in a similar manner. Potassium acts upon the oil only when heated, whereby the oil becomes brown and thickens. Alcoholic potash lye also colors the oil brown. Nitric acid acts only on heating, sulphuric acid in the cold, whereby in the former case a resin is formed, and in the latter the oil becomes dark and a black body is separated.

Hop oil is adulterated with oil of turpentine and copaiva oil, the latter sophistication being difficult to recognize. Hop oil is of considerable technical importance, it being to some extent used in breweries, expecially in those working with malt extract, to give the beer its characteristic aroma and also durability. It is also employed to increase the aroma and flavor of old or damaged hops. Though all volatile oils seem to possess the property of checking the process of fermentation, hop oil has this property in a very high degree.

## *Piperaceæ.*

*Pepper oil, oleum piperis (Pfefferöl,* G.; *essence de poivre,* F.) The unripe berries of *Piper nigrum,* L., the pepper plant, are brought into commerce as *black or common pepper,* while the ripe fruit of pepper which is immersed in water and with the hands deprived of the epicarp and sarcocarp, is known as *white pepper.* Pepper contains a volatile oil, which is obtained by distillation with water. The yield from black pepper amounts to 1.5 to 3.2 per cent., usually 1.7 to 1.9 per cent., and from white pepper to 1.4 to 1.6 per cent., according to more recent data to 2 to 2.3 per cent. Pepper oil is yellowish, limpid, and of a very pungent, pepper-like odor and taste. Sometimes it is greenish, but according to Eberhardt, this color is not due to chlorophyl nor to copper salts. The oil remains clear and liquid at —4.0 F. Specific gravity 0.87352 at 59° F.; boiling point between 332.6° and 356° F. By exposure to the air the oil becomes darker, but may be again obtained water-clear by rectification with water. It is not miscible with alcohol of 0.83 specific gravity, but becomes so by the addition of a little ether. It is slightly levo-rotatory. According to older statements the oil has the composition of oil of turpentine ($C_{10}H_{16}$). L. A. Eberhardt, however, made the following observations: The portion of the oil passing over below 348.8° F. yields, when treated with nitric acid and methyl alcohol, a crystallized terpinhydrate, $C_{10}H_{20}O_2 + H_2O = C_6H_8(OH)_2C_3H_7CH_3 + H_2O$, which commences to fuse at 219.2° F. and becomes liquid at 237.2° F., water being separated. From the fraction boiling at 348.8° to 356° F., a terpentetrabromide crystallizing in white lustrous laminæ and fusing at 251.6° to 253.4° F. could be obtained. By the formation of the nitrite the presence of phellandrene, $C_{10}H_{16}$, has been established. The use of pepper oil is a very limited one.

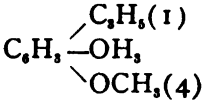
The distillate from *Piper longum*, L. is a thickish pale green oil with an odor reminding one at the same time of pepper and ginger, and a mild taste. Specific gravity 0.861 at 59° F.; boiling point between 482° and 572° F. Yield, 1 per cent.

*Betel oil* (*Betelöl*, G.) This oil is contained in the leaves of *Piper Betle*, L., = *Chavica Betle*, Miq., the betel, a climbing shrub indigenous to Java, where it is called *Sirih.* The leaves have a burning, aromatic and bitter taste, and are largely employed by the natives for chewing with slaked lime and scrapings of the areca nut.

Betel oil from fresh leaves is obtained by distillation with steam. It is slightly greenish, clear, of an agreeable powerful odor of peppermint and lemon with a creosote-like by-odor, and very pungent taste. On exposure to the air it gradually becomes golden-yellow to brown. It is slightly levo-rotatory and has a specific gravity of 0.969 at 59° F., and of 0.959 at 80.6° F. Strong potash lye colors the oil brown to purple-red, and brings about ⅓ of the volume of the oil into solution, from which by sulphuric acid a phenol is separated. This phenol is para-allylphenol, $C_6H_4\begin{matrix}-\ OH(1)\\-\ C_3H_5(4)\end{matrix}$ and is called *chavicol.* The latter boils between 456.8 and 460.4° F., and has a specific gravity of 1.03 at 59° F. It is a clear colorless liquid of an agreeable, strong betel odor, quite soluble in water and readily in potash lye. It remains liquid at —13° F., and its destructive effect upon bacteria is five times as rapid as carbolic acid, and twice as strong as eugenol.

Besides chavicol the oil of fresh betel leaves consists of sesquiterpene ($C_{15}H_{24}$) and various terpenes ($C_{10}H_{16}$), and oxygenated bodies (cineol?) the nature of which is not definitely known. Chavicol is the essential constituent to which the properties of the oil are due.

Betel oil from dry leaves is chiefly prepared by Schimmel & Co., of Leipsic. It is a slightly brown to deep brown liquid with an odor of dry tea leaves and a specific gravity of 1.024 at 59° F. By strong potash lye it is colored somewhat darker, and 70 to 75 per cent. of its volume passes into solution. Sulphuric acid separates from the solution a phenol which is an isomeric eugenol, called betelphenol, of the formula



$$C_6H_3\begin{cases} C_3H_5(1) \\ OH_3 \\ OCH_3(4), \end{cases}$$

hence, according to Eykmann, an ortho-methoxyl chavicol. The latter forms an oily, colorless, very refractive liquid of a cinnamon-like odor. It boils between 489.2 and 491° F., has a specific gravity of 1.067 at 59° F., and is very sparingly soluble in water. By treatment with an excess of 32 per cent. potash lye an oily potassium salt is separated. The saturated aqueous solution is not colored by chloride of iron, but the alcoholic solution is colored deep blue. The residue of 25 to 30 per cent. by volume of the oil remaining after the solution of the phenol yields, below 392° F., only a few drops of distillate, and between 392° and 482° F. very little, the principal mass passing over between 482° and 527° F. This portion contains cubebin, $C_{15}H_{24}$, of an agreeable tar-like odor, which yields a crystalline dihydrochloride melting at 242.6° to 244.4° F. The oil, therefore, consists of 70 to 75 per cent. of orthomethoxychavicol, $C_9H_9O(OCH_3)$, and 25 to 30 per cent. of sesquiterpene, $C_{15}H_{24}$. It is remarkable that the dry leaves contain only the constituents boiling at a higher temperature; at any rate the essential principle of the fresh leaves—the chavicol—is wanting in them. Schimmel & Co. are of the opinion that the reason for the varying results obtained in the investigations of betel oil from fresh and dry leaves is scarcely due only to the different condition of the leaves, but must be ascribed rather to conditions of climate and soil. They found in an oil from fresh leaves "betelphenol" and another phenol.

*Matico oil, oleum matico.* The leaves of *Piper angustifolium*, Ruizet Pavon = *Arthante elongata*, Miq., the matico, a shrubby plant indigenous to tropical America, contain a volatile oil which is obtained by distillation with water. The leaves are cut up, moistened with water, allowed to repose for several hours, and then distilled. The oil separating slowly from the water, two receivers arranged in the form of steps have to be used. The yield is about 2.7 per cent. The oil is yellow-brown, smells like the plant, and possesses a pungent taste of oils of turpentine and peppermint. Specific gravity 0.953 at 59° F. Matico oil is readily soluble in alcohol and ether; it is slightly dextro-rotatory, and distils mostly over at 392° F. On exposure to cold it separates a camphor fusing at 217.4° F.

The oil is adulterated with alcohol, as well as with oil of turpentine. It is used to a limited extent in medicine.

*Cubebs oil*, *oleum cubebarum* (*Kubebenöl*, G.; *essence de cubèbe*, F.), from the fruit of *Cubeba officinalis*, Miq. The cubebs are coarsely ground and then distilled with water. The oil has been also prepared by percolating the ground cubebs with light petroleum-ether, permitting this spontaneously to evaporate, and then subjecting the residue to distillation. The product was entirely free from all odor of petroleum. About 3 pounds of essential oil were obtained from 25 pounds of cubebs, and about 20 ounces of resin, fatty oil, etc., were left in the still. The oil, when recently distilled, is slightly greenish, but becomes colorless by rectification. It has the odor of cubebs and a warm, aromatic, camphorous taste. It is lighter than water, its specific gravity ranging from 0.920 and 0.936 at 59° F.; it boils at from 482° to 500° F. It mixes with glacial acetic acid in all proportions. Iodine has but little effect upon the pure oil, giving it a violet color without perceptible reaction. Treated with nitric acid it becomes opaque, and, on heating, the mixture changes to a pale red.

In a fresh state oil from fresh cubebs contains smaller quantities of dipentene, $C_{10}H_{16}$, further larger quantities of sesquiterpene, $C_{15}H_{24}$, in two modifications, one of which boils at 525.2° to 527° F., and yields a dihydrochloride, while the other has a different boiling point and does not give hydrochloride. Old cubebs oil, besides, contains a cubebs camphor, $C_{15}H_{24},H_2O$ = sesquiterpenhydrate.

Cubebs oil is used in medicine and in the manufacture of liqueurs.

### *Chenopodiaceæ.*

*Mexican oil* (*Mexikanisches Traubenkrautöl*, G.), is obtained by distillation from the herb of *Chenopodium ambrosoïdes*, L., known as Mexican tea, a plant indigenous to South America and Mexico, and possessing an aromatic odor. The volatile oil obtained from the plant is colorless, has a specific gravity of 0.9, and boils at 356° F. In taste it resembles peppermint oil.

## IV. *Volatile Oils of the Polycarpicae.*

To the tribe of *Polycarpicae* belongs a series of plants of great importance for obtaining volatile oils, the plants of the families of *Lauraceae* and *Myristicaceae* deserving special mention. The families of plants to be considered here comprise the *Lauraceae*, *Myristicaceae*, *Anonaceae*, *Magnoliaceae Monimiaceae* and *Ranunculaceae.*

All the oils to be mentioned here contain oxygen; in most of them an oxygenated body even forms the chief constituent, for instance *cineol* in bay oil, *safrol* in sassafras oil (as by-constituent in the oils of cinnamon root, camphor and star anise) *cinnamaldehyde* in the oils of cassia and Ceylon cinnamon, *eugenol* in the oils of cinnamon root and cinnamon leaf, *anethol* in star anise oil. Only the oils of mace and nutmegs consist chiefly of terpenes, but the presence in them of an oxygenated body—myristicol—has also been established. The oils of the same family of plants in some cases closely resemble one another, while in others they show a marked difference. For instance, the oils of bay berries and bay leaves, of mace and of nutmegs, are chemically identical; the oils of cassia and cinnamon are also identical, but the oils from cinnamon leaf and cinnamon root are different. Finally, attention may be drawn to the interesting fact that several of the oils belonging here contain acids or their esters; thus, lauric acid occurs in the oils of laurel and pichurim beans, cinnamic acid in the oils of cassia and Ceylon cinnamon, benzoic acid in cinnamon leaf oil, and perhaps also in cinnamon root oil. Esters occur in the oils of cassia and Ceylon cinnamon, in the oils of cinnamon leaf, cinnamon root and ylang-ylang.

Most of the oils of the *Polycarpicae* are fragrant, and are used partly in perfumery, partly in medicine, and even in a manner similar to oil of turpentine.

### *Lauraceæ.*

*Bay oil or laurel oil, oleum lauri* (*Lorbeeröl*, G.; *essence de laurier*, F.). The bay tree, *Laurus nobilis*, is indigenous to the Levant and grows wild in the countries bordering the Mediterranean. The berries (*fructus lauri*) yield bayberry oil, while the leaves (*folia lauri*) are used, on the one hand, as a spice, and on the other, for the preparation of bay leaf oil. The two volatile oils do not materially differ, though that from the leaves possesses the finer aroma. The commercial oil is chiefly prepared from the berries. The dry berries are comminuted, soaked in water and finally subjected to distillation with steam. The yield from berries is from 0.8 to 1 per cent., and from leaves 2.4 per cent.

Crude bay oil is of a greenish-yellow color, somewhat viscous and of an odor reminding one of laurel and oil of turpentine. Specific gravity 0.932 at 59° F. Boiling commences at 338° F. and slowly rises to 392° F. The oil solidifies at above 32° F., and has a bitter taste. The rectified oil is colorless to slightly yellowish, has a specific gravity of 0.914, and solidifies at 53.6° F. It has a peppermint-like odor, distils mostly over at 338° to 347° F., is very mobile, and insoluble in water. According to Mierzinski, the oil has a specific gravity of 0.932, and is soluble in two parts of alcohol.

Bay oil from the berries consists chiefly of cineol, smaller quantities of levo-pinene, as well as of sesquiterpene and lauric acid being also present.

Of reactions deserve mention: Dark brown coloration by sulphuric acid; light brown coloration by nitric acid; when heated, black coloration and subsequent decomposition. Bay leaf oil has been examined by O. Wallach. The crude oil is levo-rotatory and has a specific gavity of 0.924 at 68° F. From 316.4° to 356° F. it yields a fraction in which pinene is found, the chief mass, however, consisting of cineol. The fraction passing over above 356° F. has not been thoroughly examined.

Bay oil is chiefly adulterated with oil of turpentine; by adding to such adulterated oil sulphuric acid and then alcohol, a raspberry-red coloration is formed.

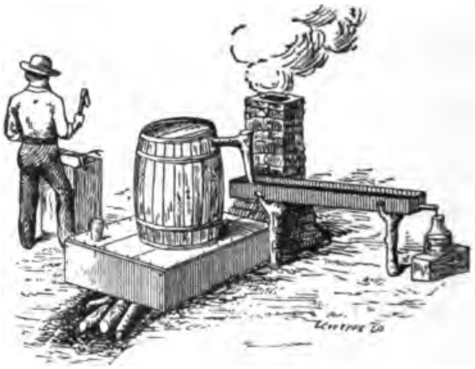
In Asia bay oil is used as an external and internal remedy. It is also said to be an excellent protection against flies and other insects which cannot bear the odor of it. It is also employed in the manufacture of soap and of liqueurs.

*Pichurim oil, oleum pichurym* (*Pichurimöl*, G.) is derived from the so-called pichurim beans, which are the cotyledons of *Nectandra* (*Ocotea*, Martius) *Puchury major* and *N. Puchury minor Nees*, two species of *Lauraceæ* indigenous to Brazil. Pichurim beans yield, according to A. Müller, 0.7 per cent. of a yellow green oil possessing the peculiar odor of the beans. It is sparingly soluble in alcohol, but readily so in ether. Bonastre, on the other hand, claims to have obtained a dirty-white oil of a slight odor and pungent bitter taste, which turns brown on exposure to the air. Both authors state having used beans of *N. P. minor*. According to Müller the oil commences to boil at 356° F., and up to 374° F. yields a colorless distillate which, when rectified at 302° F., possesses a penetrating sharp odor of *Marum verum*. The composition of the oil corresponds about to the formula $C_{14}H_{22}O_3$ (86.23 per cent. C; 11.29 per cent. H, 2.37 per cent. O). Probably both portions consist of a hydrocarbon which has suffered oxidation. The fraction above 482° F. showed after rectification a boiling point of 455° to 464° F., possessed a yellowish green color and an odor of the beans. The composition corresponded to the formula $C_{19}H_{29}O$. Finally between 509° and 518° F., quite a considerable quantity of a deep dark blue oil possessing a rancid odor passed over. In rectifying a few drops of a colorless oil first passed over. On treating the residue with soda lye a grayish green mass holding fine silky needles in suspension was formed. On heating a blue oil was separated which, after purification, boiled at 491° to 509° F. and, when subjected to distillation, formed a blue vapor. Its composition corresponded to the formula $C_{16}H_{68}O$ (86.69 per cent. C; 11.10 per cent. H; 2.81 per cent. O). The alkaline fluid finally yielded a fatty acid of the formula $C_{11}H_{23}COOH = C_{12}H_{24}O_2$, hence, lauric acid. Lower fatty acids, for instance, valerianic acid, also appear to be present in small quantities.

*Sassafras oil, oleum sassafras* (*Sassafrasöl*, G.; *essence de sassafras*, F.) is obtained from the sassafras tree, *Sassafras officinale*. The roots, together with the adhering bark, are cut into chips, 10 bushels of which yield on an average about 4 lbs. of oil. Sassafras is one of the most widely distributed trees of North America, being found in Canada, in all the United States east of the prairies, beyond the Mississippi, and in Mexico. In the north beyond the forty-second degree of latitude, near the Atlantic, it is only a shrub in size, but it exists as a tree in upper Canada 20 to 30 feet high. In the Middle States it attains, in favorable positions in good soil, a height of 30 to 40 feet and a diameter of 12 to 18 and even 24 inches, but such trees are now rare. Further south, especially in Virginia and the Carolinas, it attains a greater height and size. The largest amount of oil distilled is within sixty miles of Baltimore, which is the principal depot for its commerce. Considerable quantities are extracted in York and Lancaster counties, Pennsylvania and in some parts of West New Jersey isolated distillers, some of whom are colored men, prosecute a small business in this line. In some of the interior counties of North Carolina the sassafras tree grows in abundance, especially on worn-out lands, where it is usually found in dense thickets of small shrubs. Many primitive establishments for the manufacture of the oil may be seen there in operation. Mr. Thomas C. Harris, of Raleigh, North Carolina, in a communication to the "Popular

Science News," gives the following description of the apparatus used and method of distillation: The apparatus is so exceedingly rude and primitive as to appear ridiculous to most observers, but the product is of good quality and constitutes a profitable industry. The usual style of still is shown in Fig. 296, and may be briefly described as a short trench in the ground ending in a low flue or chimney. Over this trench, which serves as a fire-place, is placed a closed wooden box for the generation of steam. It is provided with a sheet-iron bottom and an auger hole on the top through which water is poured. An ordinary barrel stands endwise on top of the steam-box and has several holes bored through its bottom and also through the top of the steam-box, allowing steam to pass freely up through the barrel. A lute of clay is used to close the joint between the lower end of the barrel and the steam-box as well as the cover of the barrel. Instead of a "worm," a tin pipe immersed in a trough of cold water is used, and a steam connection with the barrel is generally made by an elbow branch of wood bored out with an auger. The root is dug and washed free from dirt, and, after being chopped short and bruised with a hatchet, is ready for the still. This work is done by boys, who are paid a stated price per 100 pounds for the root ready for use.

FIG. 296.



When the barrel is filled with the roots and the cover made tight with clay the process of distillation goes on rapidly. The steam passes through the mass of bruised roots and is condensed by the tin tube into a mixture of distilled water and oil and runs into a glass vessel set to receive it. Being of different densities, the oil and water rapidly settle into two strata and one can be decanted from the other.

It is said that the operator of such a still can pay all running expenses and make a clear profit of three dollars per day. When we consider that the cost of establishing such a "factory" is less than ten dollars for the entire plant, and no chemical education is necessary on the part of the operator, the profit of the work is not to be despised.

The same outfit is used in the production of the oil of pennyroyal, which grows abundantly in the woods in many counties.

Oil of sassafras when first distilled is colorless or pale yellow, but it becomes reddish with age. Its taste is pungent and aromatic, being agreeable to most persons. Its specific gravity ranges from 1.070 to 1.090 at 59° F., increasing somewhat by age. It is readily soluble in alcohol, ether, chloroform, and mixes with other fixed and volatile oils.

Crude oil of sassafras is composed of about 90 per cent. of safrol, $C_{10}H_{10}O_2$, held in solution by 10 per cent. of safrene, $C_{10}H_{16}$. According to Pomeranz, it also contains 0.23 per cent. of a phenol, which is eugenol.

The pure safrol may be separated from the oil by refrigerating the fraction of oil boiling between 442.4° and 455° F. to —13° F., at which temperature the safrol is deposited in large, strongly refractive mono-symmetric crystals. Safrol has a pleasant aromatic odor, boils at 449.6° to 451.4° F., is soluble in alkalies, and not attacked by sodium. Its specific gravity in the crystalline state is 1.245, and in the liquid state, 1.104 at 59° F.; it is then optically inactive. Safrene, on the other hand, is dextro-rotatory, its specific gravity is 0.834, and its boiling point 314.6° F.

Sassafras oil shows very decided reactions. With iodine it assumes a yellow coloration under the evolution of slight gray vapors. By strong heating with potash lye it is converted into a dark-colored, viscous, resinous mass. The test with sulphuric acid and alcohol is most characteristic. By adding sulphuric acid the mixture becomes moderately heated and almost black; by adding alcohol a deep-red fluid is obtained which will stand a considerable further addition of alcohol without losing its coloration. Sassafras oil is frequently adulterated with oil of turpentine, which is, however, readily detected by the energetic reaction, and by distilling a sample of the suspected oil. It is chiefly used in medicine, and especially in domestic practice, being reputed alterative, sudorific, and diuretic. It is also employed to some extent in perfumery and for the adulteration of other oils, especially oil of peppermint.

In Germany, sassafras oil has been almost entirely supplanted by saffrol, which, in 1885, was discovered by Messrs. Schimmel & Co., of Leipsic, in the oil of the camphor tree, *Cinnamomum camphora*, and they now separate it out in a state of absolute purity from that oil, and to an extent which probably exceeds the entire distillation of crude sassafras oil in the United States.

*Cassia oil*, or *oil of China cinnamon*, *oleum cinnamomi cassiae (Gemeines Zimmtöl*, G.; *essence de canelle de chine*, *de cassia*, F.) Cassia or China cinnamon is derived from *Cinnamomum aromaticum*, Nees (*Laurus Cassia*, Bl.) In the very interesting pamphlet, " Report of a Journey to Kwang-Si," by H. Schroeter, Hong-Kong, 1887, the writer, from personal experience, reports as follows upon the manufacture of cassia oil:

"The shrubs destined for the production of the *Cassia lignea* proper are partly stripped during the summer months of their minor branches and exceptionally juicy leaves. They are then conveyed in huge bundles into the valley, where they are boiled in large vessels. From the aromatic juice thus obtained the esteemed cassia oil is recovered by means of a most primitive distilling apparatus. As the Li-kin stations on the road to Canton levy an excessive duty upon the oil, in addition to that exacted by the Imperial Customs, the oil is carried in tin cans across the hills to Pakhoi, and thence transported, via Macao, to Hong-Kong, instead of reaching Canton by the water-way intended by nature for its conveyance."

The original boxes as received in Hamburg contain each four tin cases holding 16½ lbs. each. Cochin China also produces cassia oil. Schimmel & Co., in their report of 1890, state that the Chinese bring into commerce two kinds of cassia oil, one from the leaves and the other from the roots of the plant.

When fresh, cassia oil is pale yellow, later on, yellowish to brownish, quite thickly-fluid, of an agreeable aromatic odor, reminding one of cinnamon, and a taste at first sweet and then biting. Its specific gravity ranges from 1.055 to 1.061 at 59° F.; boiling point, 485.9° to 489.9° F. According to G. Heppe, cassia oil when kept exposed to the air, undergoes considerable alteration, so that when subjected to distillation there remains behind 20 per cent. of residue, consisting of cinnamic acid and polymerized cinnamaldehyde. On the other hand, when stored in a closed vessel the oil may be kept for a long time without injury to its quality. Even oil over 30 years old remains limpid, and, when distilled with steam, leaves behind only 6 to 7 per cent. of liquid residue. The oil solidifies below 32° F., and then fuses again at 41° F. It possesses at the utmost slight rotatory power, but is highly refractive. It is readily soluble in alcohol. It reddens litmus paper.

Cassia oil consists chiefly of cinnamaldehyde, $C_9H_8O = C_6H_5C_2H_2COH$ mixed with a little terpene, $C_{10}H_{16}$, and resin. By reason of the oxidation of the aldehyde, old oil also contains cinnamic acid, $C_9H_8O_2 = C_6H_5C_2H_2COOH$. According to Schimmel & Co., cassia oil contains 72.9 to 89.4 per cent. of cinnamaldehyde; further acetic ether of cinnamyl $C_6H_5C_2H_2CH_2 — CO_2CH_3 = CH_3COO(C_9H_9)$, very likely acetic ether of phenylpropyl, $CH_3COOC_6H_4C_3H_7$, and possibly sesquiterpenes and polyterpenes, but no terpene. Free cinnamic acid, though only in small quantity, is found in fresh cassia oil.

Of the chemical reactions shown by cassia oil may be mentioned: It slowly dissolves in iodine without perceptible heating, and is changed thereby into a thickish, yellowish-red-brown mass. According to Williams, it absorbs 72 to 75 per cent. of iodine. It also absorbs 9.6 to 10.2 per cent. of caustic potash. Hydrochloric acid, as well as ammonia, yields crystalline combinations; the same applies also to acid potassium sulphate, these being the ordinary reactions of all aldehydes. A. Ihl draws attention to the fact that with alcoholic phloroglucin solution and quite a quantity of hydrochloric acid, cassia oil yields a strong dark-red coloration, and with alcoholic resorcin solution and hydrochloric acid, a cinnabar-red coloration. Alcoholic aniline solution and hydrochloric acid color the oil yellow, especially when heated, and $\alpha$-naphthylamine solution colors it orange-yellow.

Most of the cassia oil brought into commerce is adulterated, sophistication being effected in China. A good quality of oil should have a specific gravity of 1.05 to 1.07 at 59° F., and when subjected to distillation, yield 90 per cent. distillate. The residue should at least remain thickly-fluid and amount to not more than 6 or 7, or at the utmost to 10 per cent. of the oil. The oil should contain not less than 75 per cent. of cinnamaldehyde, which may be determined by separating the aldehyde by $KHSO_3$, and weighing the residual liquid portion. By adding to 4 drops of cassia oil 4 drops of fuming nitric acid, cooling at the same time with ice water, acicular crystals or laminæ are separated on shaking, which is not the case with adulterated oil.

The principal adulterants used are resin and petroleum; the resin increases the specific gravity of the oil, which is equalized by the addition of petroleum. Clove oil is added. Oil thus adulterated does not solidify when treated with concentrated potash-lye, as is the case with pure cassia oil. Finally, cedarwood oil and copaiva oil are used as adulterants. According to Hager, copaiva balsam may be detected by shaking with ligroïn, cassia oil not dissolving in it. However, the only sure method for establishing the value of a cassia oil is the determination of the control of cinnamaldehyde. For this purpose 75 grammes of the oil are mixed in a good-sized flask with 200 grammes of a boiling 30 per cent. solution of bisulphite of sodium. The acid sulphite compound separates in the form of a coagulating precipitate. It is shaken vigorously and allowed to stand a short time. (Oils rich in aldehyde usually develop considerable heat, which must be reduced by addition of cold water.) Then about 200 grammes of hot water are added, and the whole, with frequent shaking, is warmed in a water-bath until the combination of the aldehyde with the bisulphite of sodium is entirely dissolved and the non-aldehydes, as an oily layer, float on the solution of the aldehyde salt. It is now allowed to cool, shaken up twice with ether, first with about 200 cubic centimetres, and then with 100 c.c. The ethereal extracts of non-aldehydes are separated off, brought together and filtered into a good-sized beaker, previously weighed, in which is placed a spiral-shaped platinum wire. The ether is evaporated as quickly as possible by putting the beaker in hot water. As soon as the liquid ceases to foam up when moved around, it is allowed to cool and weighed. Then the beaker is again placed in the water-bath for 10 minutes, weighed again after cooling, and so on, until the difference between two weighings is not more than 0.3 gramme. The last but one is then taken as the correct weight. The weight of non-aldehyde's thus obtained being subtracted from that of the cassia-oil used, the remainder gives the amount of cinnamaldehyde.

By repeating the experiment upon a second portion, the variation in result (if the directions are closely followed) generally amounts to a few tenths of 1 per cent., rarely to 1 per cent., which in practice is more than sufficient. The other constituents of cassia oil, including eventual adulterations (resin, petroleum, fatty oil, etc.,) are not effected by the reagent used. and, by shaking up with ether, can with ease be entirely separated from the watery solution.

The cinnamic acid found in every cassia oil is determined in

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this process as cinnamaldehyde. As, however, even in very old oil, not so much as 1 per cent. of cinnamic acid was found, the error is insignificant.

Cassia oil is chiefly used in perfumery and for flavoring liqueurs. Medicinally, it is employed as an addition to tooth powder, and has also been recommended in cholera. Used internally in larger quantities it has a poisonous effect.

*Cinnamon oil, oleum cinnamomi (Ceylon Zimmtöl,* G., *essence de canelle de Ceylon,* F.) The cinnamon tree, *Cinnamomum zeylanium,* Breyn, s. *Laurus Cinnamomi,* L. is cultivatnd in Ceylon as well as in other tropical countries, for instance, Java. Its bark forms the cinnamon of commerce, the inner bark of the young branches being of an especially fine quality. The chips and inferior pieces of the bark are reduced to coarse powder, macerated with sea-water or saturated solution of common salt for two days, and then subjected to distillation. The yield of oil is about 0.5 per cent.; according to C. F. Ballard, 0.662 per cent. from the bark, and according to Schimmel & Co., 0.9 to 1.25 per cent.

Fresh cinnamon oil is colorless, becoming golden yellow to brownish by age. It is thickly-fluid, and possesses an agreeable aromatic odor of cinnamon and a taste of intense sweetness. Its specific gravity ranges from 1.025 to 1.031; boiling point, 464° to 465.8° F. According to C. F. Ballard, the specific gravity of the best quality of oil from the bark is 1.019, while the oil from chips, bark of the root and other material of less value possesses a specific gravity ranging from 1.025 to 1.045. Java cinnamon oil is, according to Mierzinski, of somewhat inferior quality, and does not possess such a lasting sweet taste. In water cinnamon oil is quite soluble, and readily in alcohol. It is optically inactive.

The principal constituent of cinnamon oil is cinnamaldehyde $C_6H_5C_2H_2COH$; further it contains a small amount (4 to 8 per cent.) of eugenol, $C_{10}H_{12}O_2$, a strongly refractive liquid of specific gravity 1.063, and boiling point of 477.5° F., which smells and tastes of cloves.

Cinnamaldehyde may be prepared synthetically as follows: Allow a mixture of benzaldehyde 10 parts, acetaldehyde 15, water 900, and 10 per cent. solution of caustic soda, 10, to stand for 8 or 10 days at a temperature of 86° F. Frequently agitate the whole, and finally extract with ether.

Cinnamon oil, generally speaking, shows the same chemical reactions as cassia oil. Iodine, however, dissolves more rapidly and with a considerable increase in the temperature, the amount absorbed being, according to Williams, 105 to 108 per cent. The absorption of caustic potash amounts to about 5 per cent. Nitric and hydrochloric acids and ammonia give the same reactions as with cassia oil. Potash lye is without effect, on heating with solid caustic potash, hydrogen is evolved and a potassium salt formed.

Genuine cinnamon oil is adulterated with inferior qualities of cinnamon oil, especially with cassia oil, further with alcohol, chloroform, oil of cloves, etc. The adulteration with cassia oil is difficult to detect. Mr. J. Woodland* made experiments to determine a ready method of distinguishing between the two oils. It was found that if 1 part of nitric acid of 1.36 specific gravity be added to 2 parts of oil of cinnamon, and the mixture shaken, a bright orange-colored liquid is first obtained, on the surface of which floats an orange resinous substance, which slowly becomes deeper in color until a beautiful cherry-red color is visible, by which time it has changed to a liquid that floats on a lighter-colored substratum, which also in a short time becomes nearly of the same tint; bubbles then commence to appear, and shortly afterwards spontaneous ebullition occurs, with the evolution of nitrous fumes and vapors of benzoic aldehyde. By the time this ebullition has ceased, the amber-colored liquid commences to clear itself, and finally a clear amber liquid is left with orange globules floating on the surface.

If the same experiment is repeated with *oil of cassia*, a dirty-green, supernatant resinous mass, slowly turning to brown, is seen floating on a yellowish liquid, and no further change is undergone; if a large excess of the acid be added after the first addition, the resinous mass changes to a deep reddish-brown, and the subnatant liquid takes a cherry-red color. The same reaction occurs if a large excess of nitric acid be added to oil of cassia at first; but in neither of these cases is there any spontaneous ebullition or evolution of the nitrous fumes and benzoic aldehyde vapors.

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* *Year-Book of Pharmacy*, 1881.

If *oil of cassia* is mixed with *oil of cinnamon*, the reaction takes place as with oil of cinnamon, but more tardily according to the amount of oil of cassia present, and at the end of the process a turbid subnatant liquid is seen instead of a clear one.

However, according to Schimmel & Co., the only sure method for testing cinnamon oil is as follows: 1. Cinnamon oil, when allowed to fall drop by drop into cold water, should sink. 2. When placed upon the tongue, it should immediately produce a taste of intense sweetness, exceeding the sweetness of sugar.

Cinnamon oil is used in perfumery, in the preparation of liqueurs, and for medicinal purposes.

*Cinnamon leaf oil, cinnamon flower oil, clove-bark oil and cinnamon root oil.* There exists some confusion regarding the designation of these oils. Cinnamon leaf oil is distilled from the leaves of *Cinnamomum zeylanium.* Cinnamon flower oil is derrived, according to Mierzinski, from the withered calyx of *Cinnamomum Loureirii*, and, according to Husemann, from the undeveloped flowers of the same tree. Clove-bark oil is obtained, according to Hirzel, from the bark of *Dicypellium caryophyllatum*, Nees, a Brazilian plant. Regarding the derivation of cinnamon root oil next to nothing is known. Hirzel mentions that cinnamon oils of inferior quality are also obtained from the bark of *Cinnamomum Tamala*, Nees, and from *Cassia lignea*, the latter probably belonging to the *Caesalpiniaceae.*

*Cinnamon leaf oil* (Zimmtblätteröl, G.,) is thickly fluid like castor oil, of a pale-brown color, and possesses a penetrating, aromatic taste, which is at first sweetish and then pungent. It has an acid reaction and a specific gravity of 1.053, the latter ranging, according to Ballard, from 1.04 to 1.045. Generally speaking, it resembles clove and pimento oils. It is essentially a mixture of eugenol and a hydrocarbon of the formula $C_{10}H_{14}$, smelling of cymol, a small quantity of benzoic acid, and still less cinnamaldehyde. According to Schimmel & Co., the heavy cinnamon leaf oil from unfermented leaves from the Seychelles closely resembles cinnamon root oil from Ceylon. Specific gravity, 1.06; principal constituent, eugenol.

*Cinnamon root oil* (*Zimmtwurzelöl*, G.) is limpid, and possesses an agreeable odor of cinnamon and cloves. It contains no aldehydes, but 50 to 70 per cent. eugenol, further safrol, $C_{10}H_{10}O_2$, and ester of benzoic acid, perhaps, also, free benzoic acid. The oil appears to be obtained from the root of the American cinnamon tree. From the roots of the genuine cinnamon tree the Singhalese obtain a camphor-like mass which is made into candles and burned on festive occasions.

*Clove-bark oil* (*Nelkenzimmtöl*, G.) is, according to Deite, simply a name for the two above mentioned oils, while, according to Hirzel, it is obtained from the bark of *Dicipellium caryophyllatum*, Nees, a tree indigenous to Brazil. The bark of the tree is brought into commerce under the name *Cortex caryophyllatus*. The oil smells like clove oil.

*Cinnamon flower oil* (*Zimmtblüthenöl*, G.,) is derived, according to Mierzinski, from the withered calyx of *Cinnamomum Loureirii*, Nees, and according to Husemann, from the undeveloped flowers of this tree. The yield is given as 1.5 per cent. The oil, according to Mulder, has the same composition as cassia oil, which it also resembles in its other properties, only the odor being somewhat different.

*Massoi bark oil* is derived, according to Bonastre, from a tree of the laurel family, *Cinnamonum Kiamis*, Nees, indigenous to Java, and according to Maier, from *Laurus Burmanni*, a tree indigenous to New Guinea. By distillation the bark yields two oils, one heavier and the other lighter than water. The light oil is clear and almost colorless, limpid, possesses the aromatic odor of sassafras and a pungent taste. It is readily soluble in alcohol, ether and glacial acetic acid. With nitric acid it is colored a bright red. The light oil amounts to about 5 per cent. of the entire product. The heavy oil sinks in water, tastes like the light oil, but its odor of sassafras is not quite so pronounced. Its solubility and behavior towards nitric acid are the same. At 14° F. massoi bark oil becomes pasty without appearing crystalline.

On the other hand, according to C. F. Way, the oil is probably derived from a tree of the laurel family indigenons and apparently confined to the forests of New Guinea, and is used successfully as a substitute for clove oil. The oil is limpid, perfectly clear, yellow, of an agreeable aromatic odor of cloves, and a sharp, pungent taste. It is readily soluble in alcohol, ether, petroleum-ether, and glacial acetic acid. It is dextrorotatory. Specific gravity 1.0514 at 68° F. The following constituents have been found in the oil. First, a terpene, $C_{10}H_{16}$, called *massoyene*, which is dextro-rotatory, boils at 341.6° F., and has a specific gravity of 0.8581. It yields a tetra-bromide melting at 199.4° F., a nitrosochloride $C_{10}H_{16}NOCl$, melting at 208.4° F., a liquid monohydrochloride of specific gravity 0.959, and a solid dihydrochloride melting at 122° F. When heated in a closed tube it is converted into dipentene, but when heated with iodine, into cymol. It contains the groups $C_3H_7$ and $CH_3$, two double combinations, and an asymmetrical hy drocarbon atom. The dihydrobromide melts at 147.2° F., and the dihydriodide at 172.4° F.

Another constituent is safrol, $C_{10}H_{10}O_2$, with a boiling point of 449.6° to 451.4° F. The principal constituent of the oil, however, is eugenol, $C_{10}H_{12}O_2$, (about 80 per cent.) It has a specific gravity of 1.072 at 59° F., and boils at 476.6° to 480.2° F. Finally, massoi bark oil contains small quantities of creosote-like bodies. Wallach has shown that massoyene is not a new terpene, but a mixture of a small quantity of pinene with limonene. The clove odor of the oil, moreover, is claimed to be attributable, not so much to the eugenol, as to the product of oxidation or decomposition.

According to Schimmel & Co., there is a decided difference between the barks from the German colonies in New Guinea and those from the Dutch East Indics. The former contain about 7 per cent. of an oil possessing an agreeable odor of cloves and nutmegs, and the latter only traces of an oil of a disagreeable, moldy odor. The oil from New Guinea bark has a specific gravity of 1.04, boils between 392° and 572° F., and contains about 75 per cent. of eugenol. The portion of the oil insoluble in soda lye boils at 410° to 473° F., and contains among other bodies, safrol.

*Culilawan bark oil, oleum culelawan* (*Kulilawanöl*, G.,) is, according to Daryk, obtained from the bark of *Cinnamonmum Culilawan*, Nees. The bark is comminuted, moistened with water and subjected to distillation. The oil is brownish, possesses a pungent, aromatic taste, and an odor reminding one of cajeput oil and clove oil. It is readily soluble in alcohol, becomes red brown with nitric acid, and finally resinifies. By sulphuric acid the oil is colored dark-brown-red, with slight development of heat.

*Kuro-moji oil* (*Kuromojiöl*, G.,) is obtained from the wood of *Lindera sericea*, Bl., a lauraceous shrub widely distributed in Japan, where, on account of the blackish color of the bark, it is known as "Kuro-moji." Another variety of this shrub is known as Shiro-moji, on account of its white-gray bark. In Japan, an essential oil is distilled from the wood of the Kuro-moji, which has a very fine aromatic, balsamic odor, and is said to be coming rapidly in favor. According to Poleck, the oil is dark-yellow, has a specific gravity of 0.901, and contains dextro-limonene, dipentene, terpeneol and levo-carvol. Optically, it is almost inactive.

*Camphor oil, oleum camphorae* (*Kampferöl*, G.; *essence de camphre*, F.) The substance known as *Borneo camphor*, *Malay camphor*, or *borneol*, exists ready-formed in the pith cavities of the trunk of older specimens of *Dryobalanops Camphora*, Colebrook, a tree indigenous to Sumatra, and partially also to Borneo, while all parts of younger trees—their branches, leaves, etc.—contain camphor oil. Common camphor and the so-called Japan-camphor oil are obtained from *Camphora officinarum*, Nees = *Laurus Camphora*, L., a tree indigenous to China and Japan.

According to a paper read by Professor Maisch at the meeting of the Philadelphia College of Pharmacy, in October, 1890, the camphor tree is being cultivated successfully in Florida. It seems to flourish in almost any soil, and the tree grows rapidly. It is believed that in ten years the camphor industry of Florida will be very profitable. The camphor obtained from Florida trees approaches more nearly to that of Japan than to Chinese camphor, since the odor of safrol is distinctly recognizable.

*Japanese camphor oil.* In Japan, the common camphor tree is called *Kusuno-Ki*, and grows in the islands Shikoku and Kinshin, as well as in Kishin, on the southern sea-coasts. Trees about 100 years old are richest in camphor and are used for obtaining camphor and oil. The wood is distilled with water in an iron retort provided with a wooden dome, from which the vapors are conducted through a bamboo tube to the cooling apparatus. This consists of a wooden box containing seven transverse compartments, and is enclosed in a second box, through which water is allowed to flow. The vapors are conducted through all the compartments in succession by means of holes placed alternately at either end of the dividing walls. Each distilling operation lasts 20 hours. In the commencement of the work a semi-liquid mixture of camphor and oil accumulates between the walls of the cooling apparatus and floats upon the condensed water; later on, camphor alone passes over. In summer a charge of 260 lbs. of wood yields 4 to 9 lbs. of solid camphor, and 1.8 quarts of oil, and in winter, 6½ lbs. of camphor, and 0.5 to 0.8 quart of oil. The cooling apparatus is emptied every 5 or 10 days. The oil was formerly considered of no value, but at present it is gained in large quantities. The crude oil is brought into an iron kettle fitted with a cover, the latter being provided with an inlet pipe and a steam-pipe. The kettle is heated, and the vapors of the oil pass through cooling coils into a vessel, where they are again thoroughly cooled, whereby the larger amount of camphor still in solution is separated. It is then passed through a filter, and the residue subjected to pressure. The filtrate still contains camphor in solution, and is again subjected to distillation with a fresh quantity of oil. From 370 lbs. of crude oil are thus obtained 80 to 100 lbs. of solid camphor and 213 to 300 lbs. of purified Japanese camphor oil.

The yield of camphor obtained in the Florida experimental station is said to be as much as 1 lb. of crude camphor from 25 lbs. of boughs and leaves. A still of the very simplest description is used. The condenser is a straight pipe running horizontally through a trough of water; its camphor sublimes on the inner surface of the condenser, and is afterwards gotten out by standing the pipe on end and tapping the sides. A little water is put into the still with the wood to prevent burning.

Crude camphor as received from Japan and China contains from 2 to 10 per cent. of impurities, and has to be refined by sublimation. The process adopted in Philadelphia has been described as follows:*

The subliming chamber is a cylindrical iron vessel 20 feet long and 4 feet in diameter, provided with the necessary openings for filling and for the escape of the volatilized camphor. This vessel is so set that it is not touched at any point by the direct fire, and to this end rests throughout its entire length upon an arch of fire-brick, and the flame is kept under complete control by means of dampers. The condensing chamber is 30 feet long, 16 feet wide, and 11 feet high, the floor, the sides, and the arched roof being constructed of enameled brick set in Portland cement. The object of the inventor of this apparatus was to obtain the sublimate in the form of a finely pulverulent snowy mass, and this was obtained by adding one-tenth per cent. of water to the crude material before sublimation. After an operation is finished the apparatus is allowed to remain undisturbed over night, to become sufficiently cool, and the next day the sublimed camphor is removed and subjected in moulds to a pressure of 2500 lbs. per square inch in a hydraulic press. The finished product is obtained in small cakes highly compressed, weighing one ounce.

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* Oil and Drug News, March 7, 1882.

The European method of sublimation, as described by J. C. Sawer, is as follows:

The crude camphor is broken up and mixed with about 3 per cent. of quick-lime, and the same quantity of animal charcoal, both in powder. One or two per cent. of iron filings is added, and the mixture, after being thoroughly blended, is introduced through a funnel into a series of globular flasks with slightly flattened bottoms and wide necks. When the flasks are about half full, the necks are carefully freed from particles that might have attached themselves, and they are then sunk in a sand-bath, 50 or 100 together, and heat cautiously applied. The heat is suddenly raised from 248° to 374° F., and kept at that point for half an hour, so as to expel the water. The temperature is then raised to 399.2° F., and maintained at that point for 24 hours. When the crude camphor has melted, the sand is removed from the upper half of each of the flasks, and a paper cork placed in the neck. This allows of a lower temperature in the exposed part, and the vapor of camphor not being allowed to escape, condenses on the upper part of the flask as a pure cake, leaving all impurities at the bottom. Air, if freely admitted, would render the camphor opaque, but that is prevented by placing a glass bell-jar over the neck of each flask just as the vapor begins to be given off. The whole process lasts about 48 hours, and when completed the flasks are removed from the sand bath and cold water sprinkled on them. They are thus broken, and a large cake of refined camphor, 10 or 12 inches in diameter, and 3 inches thick (weighing 9 to 12 lbs.) is removed from each flask. The quicklime retains the resinorempyreumatic oil; the iron fixes any sulphur that may be present, and the charcoal removes coloring matter. Sand is sometimes mixed with the crude camphor to allow of a more uniform escape of vapor and thus save "bumping," or the sudden evolution of confined volumes of vapor. During the operation the temperature must be maintained uniformly at the point of volatilization. The process requires great care, for in addition to the very inflammable nature of the vapor, if too much heat be applied, the sublimate would remelt and fall back again to the bottom of the flask, and if the heat be not great enough the camphor sublimes in loose flakes instead of a compact cake. After breaking the flasks, the glass fragments have to be carefully separated by hand from the adhering camphor.

*Borneo camphor oil* is obtained by making incisions in the bark of the tree, and collecting the oil which flows out as a thickly-fluid turpentine-like mass, or by boiling the comminuted portions of the tree with water.

Of the two camphor oils, Japanese camphor oil has become of most importance, immense quantities of it being imported into Europe, principally into Germany. It is there refined, the camphor being separated by a freezing process, and the lighter oil distilled off. The heavier oil is said much to resembles the natural oil of sassafras, and is also used as a perfume of soap. After safrol has been separated from it, a certain quantity of eugenol (identical with the heavy portion of oil of cloves) is obtainable. Quite lately safrol has also been added in considerable proportion to the denaturalized olive oil used in greasing cotton in dyeing, to soften the product. Safrol, as manufactured by Schimmel & Co., of Leipsic, has a specific gravity of 1.108.

Japanese camphor oil is, according to Lallemand, an almost colorless, light fluid possessing a strong odor of common camphor, and is dextrorotatory. According to Oishi, purified Japanese camphor oil is colorless, burns with a bright, smoking flame, and in odor and taste resembles commom camphor. The specific gravity of the crude oil is 0.959 at 59° F., and that of the purified oil, 0.895. By exposure to the air, the oil undergoes a change, and acquires thereby a yellow color. It dissolves many resins, drying oils, asphaltum, sulphur, etc.

Mr. Kikorokura Yoshida* has published the results of his studies of Japanese camphor oil, and announced it to contain:

| | |
|---|---|
| 7 per cent. of terebenthine, boiling at | 313.8° F. |
| 20 per cent. citron, boiling at | 341 — 343.5° F. |
| 22.8 per cent. camphor, boiling at | 401° F. |
| 50 per cent. camphorogenol, boiling at | 413.5 — 415.5° F. |

Schimmel & Co., have examined the investigations of Yoshida and found that the portion boiling at 413.5 to 415.5° F. is not camphorogenol but common camphor, $C_{10}H_{16}O$, safrol, $C_{10}H_{10}O_2$, and a body boiling above 447° F., which has thus far not been closely examined. Yoshida very probably did not examine common Japanese camphor oil. Eugenol, in very small quantities, has also been found in camphor oil. Safrol is obtained on a large scale by Schimmel & Co., whereby the light and heavy camphor oils of commerce are obtained as by-products, which will be referred to below.

C. T. Kingzett has investigated the changes camphor oil suffers by oxidation and finds that by dry oxidation "soluble camphor," $C_{10}H_{18}O_3$, is first formed, which, with the formation of hydrogen peroxide, is converted into camphorogenol, $C_{10}H_{18}O_2$, or bodies isomeric with this combination. The water then contains 2 to 7.5 per cent. of soluble camphor, 1 to 4 per cent. hydrogen peroxide, and small quantities of a thymol-like body, $C_{10}H_{14}O$(?) However, later on, Kingzett admitted that camphorogenol is only a solution of camphor in camphor oil. Since 1884 Messrs. Schimmel & Co. have prepared safrol from camphor oil, whereby *light and heavy camphor oils* are obtained as by-products. *Light camphor oil* boils at 347° F. and contains in the portions boiling at a low temperature, cineol, $C_{10}H_{18}O$ (5 to 6 per cent. of the light oil), further terpenes, and probably also terpineol, $C_{10}H_{17}OH$. The portions of camphor oil boiling at a higher temperature form the *heavy camphor oil*, boiling point 464° to 518° F., in which camphor, safrol, eugenol, sesquiterpene and probably terpineol also occur.

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* Jour. Chem. Society, Oct., 1885, p. 779.

Hence, camphor oil belongs to the most compound volatile oils we know of, since as its constituents four hydrocarbons and five oxygenous bodies have to be mentioned, viz.:

Levo-pinene, $C_{10}H_{16}$ (boiling point 316.4° to 323.6° F.); limonene, $C_{10}H_{16}$ (boiling point 341.6° to 343.4° F.), or phellandrene, $C_{10}H_{16}$ (boiling point 338° F.); dipentene, $C_{10}H_{16}$ (boiling point 356° F.); sesquiterpene, $C_{15}H_{24}$ (boiling point 525.2° F.) as terpenes.

Cineol, $C_{10}H_{18}O$ (boiling point 348.8° F.); camphor, $C_{10}H_{16}O$ (boiling point 399.2° F.); probably terpineol, $C_{10}H_{18}O$ (boiling point 419° to 424.4° F.); safrol, $C_{10}H_{10}O_2$ (boiling point 449.6° F.); eugenol, $C_{10}H_{12}O_2$ (boiling point 478.4° F.), as oxygenated bodies.

Finally a few chemical reactions of camphor oil mentioned by Oishi may be given. By hydrogen chloride the oil is separated into an upper clear and a lower turbid layer. At the ordinary temperature nitric acid yields an upper yellow and a lower colorless layer; when heated a reddish liquid is formed, which gradually separates into two layers, the oil having thereby become oxidized. Sulphuric acid withdraws from the oil the elements of water and converts it into a turpentine-like liquid; an excess of acid, however, carbonizes the oil. By conducting chlorine in a slow current into the oil absorption takes place, HCl being released; the oil becomes yellow. By an excess of chlorine it becomes sticky. Bromine acts in a similar manner, but when used in excess yields a red amorphous substance. Iodine is dissolved in the cold, and on heating colors the oil red. On cooling to 32° F. a semi-solid mass is separated.

According to Oishi, the oil is used as a solvent for resins and drying oils in the manufacture of varnish. A solution of aluminium palmitate in camphor oil serves for rendering paper water-proof. In China it is used as a lamp oil, while its soot is used for the preparation of the finest quality of Chinese ink. In America sassafras oil is largely adulterated with camphor oil.

*Light camphor oil*, as brought into commerce by Messrs.

Schimmel & Co., has a specific gravity of 0.895 to 0.92 and boils at 347° F. Its flashing point, at a pressure of 763 mm., lies at 112° F. It is a colorless liquid. It was brought into commerce as a substitute for oil of turpentine, for which purpose it is quite suitable, especially as it is cheaper. In the manufacture of lacquers and varnishes it is employed for mixing with boiled linseed oil, as well as a solvent for resins, especially copal. In printing houses it is used for cleaning types and electroplates, it readily removing adhering printing ink, etc. Further, it is employed for depriving bad tallow and other fats of their bad odor; it also covers the odor of bone fat extracted with benzine. When fat thus treated is saponified, the resulting soap has an odor of camphor; for 100 pounds of soap about 2 to 3 pounds of camphor oil are required. According to R. Kayser, light camphor oil is the best solvent for caoutchouc, 1 quart of it dissolving 1 oz. of best Para caoutchouc. When the solution has been passed through linen it may be mixed with oil of turpentine, turpentine lacquer and copal lacquer, without turbidity taking place.

*Heavy camphor oil* has a specific gravity of 0.96 to 0.97, and boils between 464° and 518° F. It is of a pale green color, has an agreeably mild, aromatic taste, and is miscible in every proportion with 95 per cent. alcohol. It readily mixes with fixed and mineral oils. It ignites with difficulty, has an antiseptic effect, and may be used as an addition to lacquers, as well as for perfuming fats, lubricants, etc.

*Borneo or Sumatra camphor oil.* Crude Borneo camphor oil is a reddish, somewhat viscous liquid of a strong balsamic odor, and turns the polarized ray of light slightly to the right. Its specific gravity is 0.945. The oil is composed of: Terpene, $C_{10}H_{16}$ (boiling point, 356° to 374° F.; dexro-rotatory; specific gravity = 0.86); sesquiterpene, $C_{15}H_{24}$ (boiling point about 500° F.; specific gravity, 0.9 to 0.921; levo-rotatory); oxygenated bodies, $C_{14}H_{23}O$ (of unknown nature). Borneo camphor oil is very little used.

*California bay oil.* The California bay tree, known under the different botanical names of *Oreodaphne Californica, Laurus Californica,* and latterly *Umbellaria Californica,* is a large and beautiful evergreen tree, very common to the valleys and watercourses of the coast mountains of California. Both leaf and wood have a very fragrant aromatic odor, which, when strong, as in the crushed leaf, excites the mucous surface, brings tears to the eyes, and produces headache. Mr. J. M. Stillman * has obtained an oil by distilling the leaves with steam. Sixty to seventy pounds of fresh leaves were placed in a barrel with perforated false bottom, and steam from the escape-pipe of a steam boiler forced through the mass and condensed. In order to avoid an accumulation of too large an amount of water, this was siphoned off from the bottom of the receiving bottle, the siphon dipping into a dish of water by the side of the bottle, and the overflow of the dish kept the water in the bottle at a constant level. At the end of two days there were obtained in this way 820 grammes of the oil. The oil thus obtained is of a clear yellowish or straw color, of the peculiar aromatic odor of the leaf, and a specific gravity of 0.94 at 52° F. By long standing (nearly a year) it does not thicken.

By fractional distillation the oil separates into two portions, one boiling between 338° and 374° F., the other between 410° and 437° F.; smaller quantities passing over as high as 500°. The lesser fraction is mainly composed of terpinol, $C_{20}H_{32},H_2O$, a colorless liquid of an aromatic odor, while the higher fraction contains umbellol, $C_8H_{12}O$, a colorless mobile liquid of aromatic and powerful odor, but not volatile at ordinary temperatures. Umbellol dissolves with a blood-red color in concentrated sulphuric acid, then becomes black, and is vigorously attacked by sodium or nitric acid.

*Spice Wood Oil.* Under this name Fritzsche Bros., of New York, bring into commerce an oil obtained by distillation from the branches of *Laurus Benzoin,* L.=*Benzoin odoriferum,* Nees, a shrub common in damp woods of Canada and the United States, and known as spice-bush, wild allspice, or fever-bush.

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* Am. Chem. Jour., April, 1880.

The oil has a characteristic odor, smelling somewhat of wintergreen oil and slightly of ylang-ylang oil. It appears to be mainly composed of hydrocarbons, though it contains also 9 or 10 per cent. salicylic methyl ester. Spice-wood oil is suitable for perfuming purposes. Specific gravity, 0.855 to 0.923; boiling point, 320° to 572° F.

*Paracoto bark oil* (*Parakotorindenöl*, G.). The drug brought into commerce under the name of Paracotò bark appears to be the bark of a species of laurel which grows on the river Mapiri in Bolivia. The oil obtained from the bark by distillation with superheated steam, or by extraction with ether, is colorless, neutral, of an agreeable odor, specific gravity, 0.9275 at 59° F., and boiling point 172.4° to 482° F. It is slightly levo-rotatory. In the oil have been found two hydrocarbons and three oxygenated bodies, vis.: *α-paracotene*, of a penetrating aromatic odor resembling that of oils of bitter almonds and turpentine; boiling point 320° F.; specific gravity 0.8727; $[\alpha]_D = +9.340°$; formula, $C_{12}H_{18}$. Further *β-paracotene* of a slight agreeable odor, boiling point, 338° to 341.6° F.; specific gravity, 0.8846, $[\alpha]_D = 0.63°$, formula, $C_{11}H_{18}$; *α-paracotol*, boiling point 428° to 431.6° F., specific gravity 0.9262 at 59° F., $[\alpha]_D = 11.87°$, formula $C_{15}H_{24}O$; *β-paracotol*, boiling point 456.8° F., specific gravity 0.9526 at 15° F., $[\alpha]_D = 5.98°$, formula $C_{28}H_{40}O_2$; finally *γ-paracotol*, boiling point 464° to 467.6° F., specific gravity 0.965 at 59° F., $[\alpha]_D = 0.52°$, and formula $C_{28}H_{40}O_2$. The α-cotol reminds one somewhat of cubebs oil; it is probably the mother-substance for β- and γ-cotol which are formed from it by oxidation, with the formation of water and formic acid.

## *Myristicaceæ.*

*Mace oil, oleum macidis* (*Muskatenblüten- or Macisöl*, G.; *essence de macis*, F). The nutmeg-trees, *Myristica fragrans*, Houtt. = *Myristica moschata*, Thunbg. is indigenous to the Moluccas, but is also cultivated in other tropical countries. When the nutmeg is gathered the laciniate envelope, usually called the aril, is cut off and dried in the sun, whereby its bright red color is changed to brownish orange. This dried aril forms the mace of commerce. It also contains fat oil which may be obtained by expression. The mace freed from fat oil is subjected to distillation with water, whereby volatile oil is obtained. The yield amounts on an average to 7.7 per cent., so that mace may be considered one of the parts of plants richest in oil.

Mace oil is water-clear to pale yellow, and by long keeping becomes yellow-red. It is somewhat thickly-fluid, possesses an agreeable odor of mace and a pungent, aromatic taste. Its specific gravity ranges, according to Maier, from 0.9266 to 0.947; according to Williams from 0.886 to 0.90 at 60° F., and, according to Semmler, is 0.9309 at 57.2° F. It boils, according to Maier, between 374° and 392° F., and, according to Williams, at 346° F. It dissolves in 5 parts of alcohol. Older oil is specifically heavier than fresh oil. and the former reddens litmus paper. The oil is dextro-rotatory. The product from fresh mace has a more delicate odor than that from dried mace.

According to Semmler, mace oil consists of 53 per cent. of terpenes, 15 per cent. of a mixture of terpene and myristicol, $C_{10}H_{16}O$, 9 per cent. myristicol and myristic acid, and 22 per cent. myristicin, $C_{12}H_{14}O_4$. The latter is a solid, ester-like body, melting at 86° F.

Mace oil reacts violently with iodine and, according to Williams, absorbs 221 to 224 per cent. of the latter. It absorbs also 0.67 to 0.85 per cent. of caustic potash. By nitric acid it is colored red in the cold, but on heating it is converted into a soft, yellowish-brown resin, violent reaction taking place thereby. By sulphuric acid the oil is colored brownish-red, it being thickened at the same time; alcoholic chloride of iron colors it emerald green.

Mace, before being brought into European commerce, has frequently been extracted with alcohol by the Chinese, so that it only yields a small amount of oil (4 per cent). It is, moreover, difficult to obtain good raw material; the export of oil prepared abroad (especially from Singapore) is also decreasing. Mace oil is adulterated with alcohol, foreign volatile oils, petroleum, etc. It is used for perfuming soap, in the preparation of liqueurs, and, according to Maier, as an internal remedy for gall-stones.

*Nutmeg oil* (*volatile*), *oleum nucistæ æthereum* (*Muskatnussöl*, G.; *essence de muscade*, F). The nutmeg, the seed of *Myristica fragrans*, Houtt., contains a fat and a volatile oil, the latter being chiefly obtained from inferior and broken nutmegs, not salable as spice. The broken material is comminuted, the volatile oil expelled by steam, and the fat oil obtained by subjecting the residue to pressure. By this process about 5 to 6 per cent. of volatile oil is obtained.

Volatile nutmeg oil is colorless or pale yellow, quite limpid, and has the odor and taste of nutmegs. Its specific gravity ranges from 0.8826 to 0.948, and, according to Williams, from 0.883 to 0.898 at 60° F. According to the same author it boils at 339.8 to 341.6° F. On exposure to the air it becomes darker and separates crystals. By agitation with water it separates into two oils, one lighter, the other heavier than water. The oil is soluble in 5 parts of alcohol and in all proportions in glacial acetic acid.

Volatile nutmeg oil is composed of a small quantity of cymol, $C_{10}H_{14}$; chiefly of terpene, $C_{10}H_{16}$ (70 per cent.; boiling point about 329° F.; specific gravity = 0.8533 at 59° F.; levorotatory, yields a liquid hydrochloride, $C_{16}H_{16}HCl$, which boils at 381.2° F.); further of a smaller quantity (15 per cent.) of another terpene, $C_{10}H_{16}$ (boiling point, 343.4° to 354.2° F.); next of myristicol, $C_{10}H_{16}O$; finally of a resin, $C_{40}H_{56}O_5$ (5 per cent., not volatile even at 572° F.), and myristicin, $C_{12}H_{14}O_3$.

According to Williams, nutmeg oil absorbs 202 to 206 per cent. of iodine and 0.19 to 0.35 per cent. of caustic potash. In its chemical reactions it resembles mace oil.

Nutmeg oil is adulterated by the same additions as mace oil, and is used for the same purposes. Taken internally it has an exciting and stimulating effect, especially upon the nerves and stomach. In larger doses it is poisonous. Applied to the skin it causes burning and reddening, almost like mustard oil.

### *Anonaceæ.*

*Ylang-ylang oil, oleum anonæ* (*Ylang-ylang or Kanangaöl*, G.; *essence d' ylang-ylang*, F.). This essential oil was first introduced in 1864. It is obtained by distilling the flowers of *Cananga odorata*, Hooker, natural order, *Anonaceæ*, a tree which grows in the Philippine Islands, the Straits of Malacca, and Indian Archipelago. The flowers are flocculent and drooping and of a greenish-yellow color. They yield a colorless or slightly yellowish oil of an exquisite odor, somewhat partaking of the jasmine and the lilac. The oil is chemically of interest, as it contains benzoic acid, very likely in the form of a compound ether, and Flückiger has shown that it also contains a phenol-like body and an aldehyde or ketone. The specific gravity of the oil is 0.980 at 59° F., and its boiling point from 320° to 572° F. It was formerly held at an enormous price, but at present fair to good oils may be obtained in large quantities at prices ranging from 3/6 to 8/6 per oz. The yield, according to Reymann, is quite small, 25 grammes of oil from 5 kilogrammes of flowers, and there are annually consumed in Paris, Nice, and Grasse about 200 kilogrammes (440 pounds), in London about 50 kilogrammes (110 pounds), and as much in Germany. It is extensively used in the manufacture of fine perfumes.

It may be remarked here that the flowers of another species of *Anonaceæ*, the burrow pepper, *Xylopia longifolia*, which grows in the district of Chirigui, Costa Rica, yield a volatile oil, which is said to be of a still more agreeable odor than ylang-ylang oil. Attention may also be called to the mosoi flowers introduced from Samoa. They smell like ylang-ylang flowers and are derived also from *Cananga odorata*. It is of special interest that the oil is partially separated in stone-cells, which is very seldom the case. The flowers yield 1.2 per cent. of oil of specific gravity 0.922 and the odor of ylang-ylang oil. The bark of the same tree, known as *mosoi bark*, is also brought into commerce. It smells like galbanum.

### *Magnoliaceæ.*

***Star anise oil, Chinese anise oil or badiane oil, oleum anisi stellati*** (*Sternanisöl*, G.; ***essence de badiane***, F.) is obtained by distillation from the fruit of the star anise, ***Illicium anisatum***. The tree is cultivated in the mountainous regions of Annam; it grows very slowly during the first ten years, and up to that time produces very little. A full-grown tree yields about 400 pounds of fruit. In Annam the process of distillation is as follows: About 20 pounds of star anise and water are placed in an iron pan. Upon this is placed, bottom upwards, so as to form a cover, a second iron pan pierced with a circular opening. Over the opening in this pan is placed an earthen vessel having three small orifices in the lower part, which allow the access of the vapor into this vessel. These orifices are covered on the inside of the vessel by small ear-shaped hoods that have for their object to throw the vapor upon the side of the vessel. Lastly, this earthen vessel is covered with an iron pan, which performs the part of a refrigerator, and into which a continuous current of cold water is passed by means of a bamboo pipe from a vat placed near the apparatus. The joints of the first two iron pans are luted with a mastic and the joint of the earthen vessel with the refrigerant by means of rag bandages. The vapor reaching the earthen vessel is condensed when it strikes upon the bottom of the pan holding the cold water and falls into a small circular trough running round the bottom of the vessel, from which it escapes through a small pipe. The small pipe opens out into a bamboo which conducts the products of distillation into a closed tinned vessel full of water, where the oil, rising to the top, drives out a corresponding quantity of water. The yield is said to be 2.46 per cent. The oil is carried in clay jars to the city of Lank-Son, where it is put up in tin cans. France is endeavoring to concentrate the entire commerce of star anise oil in Havre or Marseilles. In Europe the fruit is comminuted and distilled with steam; the yield of oil is 3 to 5 per cent.

Star anise oil is colorless to yellowish, it becoming darker

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with age. In taste and odor it resembles aniseed oil; it is more limpid than the latter and separates stearoptene only below 35.6° F. Its specific gravity is 0.982 at 59° F. It is optically inactive or very slightly levo-rotatory. It dissolves readily in alcohol and ether.

Investigations made by Messrs. Schimmel & Co.* into the nature of star anise oil throw some light upon the character of the constituents of low boiling points, viz., the terpenes contained in it. The fraction in which these constituents occur boils over between 314.6° and 347° F., and contains: *Dextro-pinene*, boiling point, 314.6° to 325.4° F., optical rotation + 21°30′ (100 m.m. tube), characterized by the pinene-nitrolanine base of a melting point of 251.6° to 253.4° F. But the chief constituent is *levo-phellandrene*, boiling point, 338° to 347° F., optical rotation —5°40′ (100 mm. tube), melting point of the nitrate, 215.6° F.

By boiling, nitric acid converts star anise oil into a viscous, red balsam, while iodine changes it into a solid resin, with the development of slight heat. When heated with sulphuric acid, it is first colored dark red, and is then more energetically attacked, so that, after cooling, a blackish red-brown resin remains behind. Alcoholic hydrochloric acid colors the oil yellow-brown to brown.

Star anise oil possessing a finer odor than aniseed oil, it is preferred for perfuming toilet soaps and in the preparation of liqueurs. Otherwise it is used for the same purposes as aniseed oil.

*Shikimi oil* or *Japanese star anise oil* is derived from the fruits of *Illicium religiosum*, a tree of Japan, resembling the star anise tree and known in Japan as Shikimi-no-ki. The fruit of this tree was brought into commerce mixed with star anise, but as it contains a poisonous substance, called shikimin, the volatile oil obtained from such mixture acquired poisonous properties. The actual *shikimi oil* or *false aniseed oil* is, however, obtained from the leaves of *Illicium religiosum*. As a result of his investigations, J. F. Eijkman reports that the leaves yield 0.44 per cent. of volatile oil of specific gravity 1.006. The oil is slightly levo-rotatory. It has a weaker odor than star anise oil. It consists of eugenol and two constituents which he calls shikimol and shikimene. Shikimol is identical with safrol, $C_{10}H_{10}O_2$, and shikimene, $C_{10}H_{16}$, probably with safrene.

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* Report, April, 1893.

Alcoholic hydrochloric acid is after some time colored pale blue by the oil; ammoniacal silver solution is in a few hours reduced by it. Chloral hydrate colors the oil dirty brown-yellow, but genuine star anise oil red.

### *Monimiaceæ.*

*Boldo leaf oil* is obtained from the leaves and young wood of the Chilian tree commonly known as Boldo (*Boldoa fragrans*, Gay). According to Bourgoin and Berne, the oil is clear, reddish yellow, of a pungent taste and aromatic odor. Specific gravity = 0.9183; boiling point between 356° and 572° F. Besides substances the nature of which is not yet known, it contains an alkaloid which can be precipitated with alkalies. According to Schimmel & Co., fresh imported boldo leaves of a good quality yield 2 per cent. of oil, which possesses in a concentrated form the aromatic pepper odor of the plant, and has a mild, indifferent, herb-like taste. The specific gravity of the oil is 0.918 and the boiling point 347° to 482° F. The oil contains terpenes and oxygenated bodies. It is used in liver affections and gall-stones, and has recently been also employed in gonorrhœa, dyspepsia and rheumatism.

*Australian sassafras oil* is obtained by distilling the dried bark of *Atherosperma moschata*, R. Br., a tree indigenous to Australia and Tasmania, found also in Chili. It is pale yellow, possesses a peculiar odor, and volatilizes almost entirely at 435.2° F. It has a specific gravity of 1.0386, and is dextro-rotatory. The bark is used as a substitute for tea and is slightly purgative. It contains an alkaloid as well as a tannic acid, $C_{16}H_{18}O_3$ (Husemann).

### *Ranunculaceæ.*

*Fennel-flower oil* (*Schwarzkümmelöl*, G.), also called *nigella oil*, is obtained by distillation from the seed of the fennel-flower, *Nigella sativa*, L., a plant indigenous to Southern Europe and the Levant. The yield of oil is 0.8 to 1.5 per cent. The oil is colorless and clear, with a blue fluorescence; it is lighter than water, smells of oils of fennel and bitter almonds, and has a mild aromatic odor. It is miscible in every proportion in absolute alcohol. It has a neutral reaction. When shaken with potash lye and the mixture subjected to distillation, a water-clear oil of a slight odor passes over; from the residue sulphuric acid precipitates white flakes, which dissolve in ether and, after the evaporation of the solvent, form a butyraceous oil smelling of camphor. The distillate contains terpene, $C_{10}H_{16}$; while the oil smelling like camphor contains a body, $C_{20}H_{24}O$, of unknown constitution (Maier, Husemann).

## V. *Volatile Oils of the Dicotyledons.*

Although this group contains only two volatile oils (oils of mustard and geranium) of any importance, it is nevertheless of special chemical interest because, on the one hand, the oils in some cases do not occur ready-formed in the plants, and, on the other, they generally contain nitrogen or sulphur. The following families come here under consideration:

*Cruciferæ, Canellaceæ, Ternstrœmiaceæ, Tiliaceæ, Geraniaceæ and Tropædaceæ.*

Of the volatile oils derived from the plants of these families only the oils of canella, tea, linden flowers, and geranium belong to the fragrant oils, and of these only the latter occurs to any extent in commerce. The oils of canella and geranium contain oxygen, the chief constituent of the latter being geraniol, $C_{10}H_{18}O$, while in the former eugenol and cajeputol appear among other components. The oils of mustard, cochlearia, horse-radish, white mustard, radish, and hedge garlic contain sulphur, the oils of mustard, horse-radish and hedge garlic consisting of allyl thiocarbimide, $C_3H_5NCS$, while cochlearia oil contains isobutylthiocarbimide, $CH_3H_8NCS$, and white mustard oil oxybenzylthiocarbimide, $C_6H_4(OH)CH_2NCS$. The principal constituents of the oils of garden cress, water cress and Indian cress are nitrides, the oils of garden cress and Indian cress consisting of phenylacetonitril, $CH_2C_6H_5CN$, and water cress oil of phenylpropionnitril, $C_2H_4C_6H_5CN$. The fragrant oils of this group are lighter than water, the others heavier. The mustard oils are chiefly used for medicinal purposes, and are largely formed by fermentation from glucoside-like constituents of the plant.

### *Cruciferæ.*

*Cress oil* (*Kressenöl*, G.), is obtained from a series of species of *Lepidium* (family of *Cruciferæ*). By direct distillation with steam it is prepared from the herb of *Lepidium ruderale*, L., the wall-cress, and the herb of *Lepidium sativum*, L., the garden cress, when just done blooming, because in the seed of these two plants the oil does not occur ready formed, as in *Lepidium campestre*, R. Br., the penny-cress, but is formed only after the action of water. The seed, though rich in oil, is difficult to distil, it forming a thick paste with the water.

From the herb of the wall-cress Pless obtained the oil as follows: The herb was comminuted, mixed with water, and then subjected to distillation. Copper retorts could not be used because the oil is partially decomposed by them. The distillate formed a milky fluid, from which, after several rectifications, a yellow oil was separated. Long standing of the herb with water before distillation is injurious, because the oil may thereby be completely decomposed.

*Wall-cress oil* (*Mauerkressenöl*, G.) is heavier than water, of a peculiar, refreshing, somewhat leek-like odor, and the pungent taste of water-cress. The inhaling of the vapors causes a disagreeable sensation of dryness in the throat, and headache. The oil, when several times rectified, is colorless, but on exposure to the light soon turns yellow. In water it dissolves with difficulty, but readily in alcohol and ether. In sulphuric acid it dissolves with a red color, and separates again when water is carefully added. The oil contains sulphur.

By distillation with steam the herb of garden cress yielded, according to Trommsdorf, a distillate without a layer of oil; the oil could, however, be withdrawn from the water by benzol. The yield was 0.115 per cent.

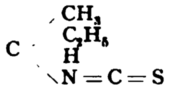
*Garden cress oil* (*Gartenkressenöl*, G.) is, according to Hoffmann, clear, transparent, and of a pale yellow color, but becomes colorless by rectification. Boiling point 439.7° F. (corrected 448.7° F.). By boiling with soda lye ammonia is evolved, while by adding hydrochloric acid phenylacetic acid is separated from the alkaline solution. Hence, the principal constituent (at least 75 per cent.) of the oil is phenylacetonitril, $C_8H_7N = CH_2C_6H_5CN$; though small quantities of a combination containing sulphur, which has not yet been thoroughly examined, are also present.

Since, according to Pless, wall-cress oil is not perceptibly altered by potash lye, it must possess a different composition from garden-cress oil.

A volatile oil may also be obtained by subjecting the leaves of *Lepidium latifolium*, L., the broad-leaved pepperwort, to distillation. The oil is heavier than water and seems to contain sulphur (Maier).

*Water-cress oil* (*Brunnenkressenöl*, G.). By distillation with steam, water-cress (*Nasturtium officinale*, L.) yields a distillate from which a volatile oil can be withdrawn by means of petroleum-ether. It is, however, quite difficult to free the oil entirely from the last traces of petroleum-ether. The yield is quite small, amounting only to 0.0067 per cent. A. W. Hofmann has subjected the oil to a close investigation. It smells of the plant, has a specific gravity of 1.0014 at 64.4° F., and boils from 356° to 536° F. The principal mass of the distillate is formed by an oil which, when rectified, boils at 488.3° F. (corrected 501.8° F.). When boiled with soda lye the oil evolves ammonia and is a nitril of the formula, $C_9H_9N$. Since by the oxidation of the nitril, phenylpropionic acid is obtained, the principal constituent of water-cress oil is evidently phenylpropionitril, $C_2H_4C_6H_5CN$. Besides this substance the oil contains hydrocarbons (very likely originated from the extractive agent), and in the higher fractions a body crystallizing in octahedrons which have not been closely examined.

*Cochlearia oil or scurvy grass oil*, *oleum cochleariæ* (*Löffelkrautöl*, G.) is obtained by distillation from *Cochlearia officinalis*, L., the common scurvy grass, a native of Northern and Central Europe, where it grows in saline soil and is occasionally cultivated. The oil is yellow, heavier than water, and very volatile. It possesses a penetrating odor and a pungent taste. It is readily soluble in alcohol. A. W. Hofmann found the boiling point of the rectified oil to be 318.2° to 320° F., and by the action of ammonia upon the oil prepared from it a beautiful crystallizing substance melting at 275° F. was obtained. As early as 1869, the analysis gave the formula $C_5H_9NS$, which corresponds to a butyl sulphocarbimide, $C_4H_9CSN$. Later on Hofmann again prepared the oil in larger quantities. The fresh herb was cut up, stirred to a thin paste with water and distilled from copper vessels. The yield was 0.034 per cent., which was, however, somewhat less than the actual amount, because a portion of the oil was decomposed on the copper walls of the still, sulphide of copper being formed. The rectified oil boiled at 321.8° to 325.4° F. and had the composition $C_4H_9CSN$. Further investigation showed it to be an isosulphocyanate of secondary butyl alcohol, hence isobutylthiocarbimide, the structural formula of which is as follows:



$$C\begin{cases}CH_3\\ C_2H_5\\ H\\ N=C=S\end{cases}$$

Hofmann succeeded in preparing the oil synthetically. Such oil has been brought into commerce, since 1889, by Schimmel & Co., of Leipsic, as a substitute for the natural oil, since there is not the slightest probability that the latter can be produced at anything like a reasonable price. A mixture of rue oil with a small quantity of mustard oil is frequently sold as cochlearia oil, which, of course, does not possess the properties of the latter. An alcoholic solution of cochlearia oil obtained by extracting the leaves with strong alcohol is used in cases of debility, scrofulous diseases, chronic catarrh and scurvy.

*Horseradish oil* (*Meerrettigöl*, G.), is obtained from the root of *Cochlearia armoracia*, L., the horseradish, and, according to Hubatka, occurs ready-formed in the root. Distillation has to be effected in a glass retort, since copper vessels cause a partial decomposition of the oil, sulphur being fixed. The oil is heavier than water and of a pale yellow color. Yield 0.052 to 0.073 per cent. The crude oil is again distilled with 4 parts of water and freed from water over calcium chloride. It is then colorless to slightly yellowish, and of specific gravity 1.01. Its odor is penetrating like that of mustard oil; applied to the skin it draws blisters. It is soluble with difficulty in water, but readily in ether and alcohol. The composition of the oil corresponds to that of mustard oil, and is hence expressed by the formula, $C_3H_5CSN$.

*Penny-cress oil* (*Täschelkrautöl*, G). By cutting up the herb or seed of the penny-cress (*Thlaspi arvense*, L.), mixing with water, and distilling, a colorless oil of a penetrating odor and pungent, alliaceous taste passes over after some time. F. Pless found the oil to be a mixture of the oils of garlic and mustard, *i. e.*, of allylsulphide $(C_3H_5)_2S$, and allylthiocarbimide, $C_3H_5CSN$. Both these bodies do not occur ready-formed in the seed, but are formed by the action of water. The alcoholic extract of the pulverized seed when evaporated leaves behind a residue which, when triturated with myrosin and water, yields mustard oil.

*Mustard oil, oleum sinapis* (*Senföl*, G.; *essence de moutarde*, F.). The seed of the black mustard, *Brassica nigra*, Kch. = *Sinapis nigra*, L., contains, besides fat-oil, a peculiar albuminous body—*myrosin* and *potassium myronate*. If, in the presence of water, these two bodies act upon each other, volatile mustard oil is formed. To obtain the latter it is best to free the seed from fat oil by expression, and to stir the comminuted press-cakes with 3 to 6 parts of water to a paste. The water should be lukewarm (not over 122° F.), since at a higher temperature the myrosin coagulates and becomes inactive. After 24 hours the mass is distilled with direct steam; the use of direct fire should be avoided. The water of the distillate contains mustard oil in solution, and may be used in mixing the press-cake for another operation. It is also of advantage to add a paste of white mustard seed, the latter being rich in myrosin. The seed of *Sinapis juncea*, L., the sarepta or Russian mustard, is now frequently used in place of the seed of black mustard. The yield from black mustard is about 0.7 to 1.0 per cent.; according to Schimmel & Co., 0.9 per cent., and from sarepta mustard, 0.52. Some time is required for the oil to separate from the water; it should be separated in a separatory funnel, and finally filtered through paper.

Mustard oil is a water-clear, mobile, strongly refractive liquid, turning yellowish with age. It possesses in the highest degree the sharp, pungent odor of mustard. It rapidly blisters the skin, and its vapor excites tears and produces even in a small quantity inflammation of the eyes. Its specific gravity is 1.018 to 1.029 at 59° F.; it boils at 298.5° F. One part of it dissolves in 50 parts of water, but readily in ether and alcohol. When heated mustard oil dissolves sulphur and phosphorus, but separates them again in a crystallized form on cooling.

Volatile mustard oil consists of allyl sulphocarbimide, $C_3H_5NCS$, mixed with small quantities of allyl carbamine, $C_3H_5CN$, and, when prepared at too low a temperature, traces of allyl cyanide, $C_3H_5SCN$.

Hager recommends the following tests for adulterations:

1. *Evaporation.*—One or two grammes of the oil are evaporated in a shallow dish at from 104° to 122° F. If pure, the oil evaporates totally in the course of 2 hours; a residue indicates the presence of fixed oils, phenol, oil of cloves, oil of cinnamon, etc.

2. *Dropping into cold water.*—Strictly pure oil sinks in water and remains clear for hours. If the drops become cloudy or milky in a minute, 1 per cent. of alcohol or amylic alcohol is present.

3. *Pure concentrated sulphuric acid.*—10 drops of the oil are mixed in a test-tube with 4 to 5 per cent. sulphuric acid; when no change of color is produced the oil is pure; a darker coloration indicating the presence of adulterations, such as fixed oils, carbon disulphide, chloroform, etc.

4. *Solution of ammoniated copper.*—The solution is made by adding ammonia gradually to a concentrated aqueous solution of copper sulphate until the precipitate is redissolved. To 10 drops of the suspected oil, dissolved in about 4 cubic centimetres of pure absolute alcohol, add 2 or 3 cubic centimetres of the copper solution, when an ultramarine-blue precipitate falls, which does not change its color if the oil is pure. If a trace of carbon disulphide is present, the precipitate turns first violet-brown and then dark reddish-brown in a few minutes. The presence of amylic alcohol, phenol, or oil of cloves, also causes a change of the color.

5. *Test for carbolic acid.*—Mix 10 drops of the suspected oil with 10 cubic centimetres of water; shake, filter 15 minutes later, and add to the filtrate 2 drops of solution of ferric chloride, when the presence of carbolic acid is indicated by a blue coloration.

6. *Test for carbon disulphide.*—This adulterant was quantitatively determined by distilling 100 grammes of the oil contained in a previously weighed glass retort, first in a water bath and afterwards in a glycerin bath. The carbon disulphide began to distill at 140° F., and continued to come over until the temperature had been raised to 357.5° F.

Mustard oil is chiefly used for medicinal purposes.

*White mustard oil.* The volatile oil of white mustard seed is not used, and is quite difficult to prepare. It is of a yellow color, possesses an extremely pungent taste, and, when applied to the skin, draws blisters; but its action is not so powerful as that of black mustard oil. It is scarcely soluble in water, but readily in alcohol and ether. The oil very probably contains also a certain amount of the cyanide combination, $C_7H_7OCN$. H. Salkowski has recently examined the oil as to its composition and constitution, and is of the opinion that it is paraoxybenzyl mustard oil. The preparation of the oil is difficult because it cannot be obtained directly from the seed, it being necessary to first prepare pure sinapine. Paraoxybenzyl mustard oil may be prepared synthetically by allowing paraoxybenzylamine to be acted upon by carbon disulphide, and treating the product of reaction with chloride of mercury.

*Radish oil* (*Rettigöl*, G.). The root and seed of *Raphanus sativus*, L., when distilled with water, yield, according to F. Pless, a turbid distillate, which by rectification furnishes a small quantity of a colorless volatile oil. The latter tastes like radish but differs from it in odor. It is heavier than water and quite soluble in it. If it be oxidized with nitric acid the product of reaction yields, with barium chloride, a precipitate of barium sulphate, so that radish oil may be considered sulphuretted.

*Hedge garlic oil* (*Knoblauchhederichöl*, G.) is obtained from the root as well as from the seed of *Erysimum alliaria*, L. = *Alliaria officinalis*, R. Br., the hedge garlic. The root cut in slices and distilled with water yields, according to Wertheim, 0.03 per cent. of volatile oil, which possesses the properties, and probably the composition, of mustard oil. On the other hand, the herb is said to yield an oil possessing the composition of garlic oil, $(C_3H_5)_2S$. F. Pless has also investigated the oil from the seed; he obtained 0.6 per cent. of oil and found it to consist of 90 per cent. mustard oil and 10 per cent. garlic oil. In another oil the latter was, however, entirely wanting.

## *Canellaceæ.*

*Canella oil* (*Kanellaöl*, G.) is obtained from the so-called white cinnamon, the bark of *Canella alba*, Murr., the white wood or wild cinnamon tree, indigenous to many of the West Indian islands, and the southern section of Florida. The bark is distilled with steam and yields 1.25 per cent. of oil, which is lighter than water and of a spicy odor. If the oil be left in ontact with strong potash lye for several days, and the mixture be then diluted with water and subjected to distillation, the greater portion of the oil passing over is lighter than water, but the last portions sink in water. The heavier portion constitutes the odoriferous part of the oil, and posseses, according to analysis, which is, however, not quite reliable, the formula $C_{27}H_{47}O_4$. The lighter portion smells almost like cajeput oil, and when distilled with water yields several fractions of varying composition: 1. Boiling point, 332.6° F.; formula, $C_{11}H_{20}O$. 2. Boiling point 356° F.; formula, $C_{15}H_{27}O_2$. 3. Boiling point 473° F.; specific gravity, 0.941; formula, $C_{12}H_{19}O$ or $C_{24}H_{30}O_2$. In distilling off the oil mixed with potash lye, a residue remains behind in the retort which, by adding sulphuric acid and subsequent distillation, yields an oil which is heavier than water and possesses the odor of cloves; its composition is that of eugenol. Hence canella oil consists of four oxygenated bodies, of which eugenol and probably cajeputol are the most prominent representatives.

### *Ternstroemiaceæ.*

*Tea oil* (*Theeöl*, G.). From tea, the young, dried leaves of *Thea chinensis*, Sims, the tea shrub, a volatile oil may, according to Mulder, be obtained. The tea is extracted with ether, the latter allowed to evaporate in the air, the residue washed with water, and distilled. The distillate is again shaken with ether and the ethereal extract again evaporated at the ordinary temperature. The yield is 0.6 to 1 per cent. The oil is of a lemon-yellow color, lighter than water, has a strong odor of tea and the property of readily decomposing and quickly solidifying in the cold. The odor is stupefying, and the oil, when taken internally in certain quantities, acts as a poison.

*Gurjun oil* or *wood oil*. Gurjun balsam or wood oil was formerly considered a variety of copaiva. It is, however derived from *Dipterocarpus turbinatus* and other species of the same genus (*D. incarnus, alatus, costatus, etc.*). The balsam is brought into commerce chiefly from Moulmein, British Burma, and forms, according to Guibourt, a dark liquid of specific gravity 0.964, and a taste and odor of copaiva. In alcohol it is completely soluble. When heated by itself or with magnesia it becomes solid. According to C. Lowe, the balsam consists of 65 per cent. volatile oil, 34 per cent. resin, and 1 per cent. water and acetic acid. By subjecting it to distillation with a small quantity of chloride of lime a blue colored oil passes over. By rectification with steam a colorless oil, boiling at 491° to 492.8° F., is obtained. It has a specific gravity of 0.918 and consists of diterpene, $C_{20}H_{32}$, or sesquiterpene, $C_{15}H_{24}$, and smells like copaiva oil.

### *Tiliaceæ.*

*Linden flower oil* (*Lindenblüthenöl*, G). From the flowers of *Tilia grandifolia*, Ehrh., *T. parvifolia*, Ehrh., the linden, 0.05 per cent. of a colorless volatile oil, is obtained by distillation with water, addition of common salt and shaking with ether. The oil is limpid, quite volatile, quite constant in the air, and possesses a strong odor of the flowers. It dissolves without heating in iodine to a brown-red liquid, and is insoluble in alcohol.

### *Geraniaceæ.*

*Geranium oil*, *oleum pelargonei* (*Geranium- or Palmarosaöl*, G.; *essence de géranium*, *essence roses d'Afrique*, *de feuille de rose géranium*, F). The various oils of geranium known as African, Spanish, French and Réunion are derived from the species of Pelargonium, the *P. odoratissimum*, Willd., *P. capitatum*, Aiton, and *P. roseum*, Ait., but the plants in actual cultivation are varieties of these pelargoniums.

French oil of geranium is obtained by aqueous distillation from the leaves and flowers of *Pelargonium radula*, natural order *Geraniaceæ*, largely cultivated in Southern France. It is colorless, occasionally greenish, yellowish, or brown, the latter being most esteemed. It boils at 421° to 428° F., and solidifies at 3.2° F. It has a pleasant odor, resembling that of oil of rose, and is dextro-rotatory.

The Algerian or African oil is distilled from the leaves and flowers of *Pelargonium roseum*, Aiton, and *P. odoratissimum*, Willd., largely cultivated in France and other countries. It greatly resembles French oil of geranium, but is levo-rotatory. Both oils are frequently used as adulterants of rose oil, and are themselves adulterated with the oils of certain species of *Andropogon*. According to Jaillard, the oils are freely soluble in 70 per cent. of alcohol. Since the fixed and most volatile oils which may be used for adulteration are sparingly soluble in that liquid, they may be detected by mixing in a test-tube 6 drops of the suspected oil with 5 cubic centimeters of 70 per cent. alcohol. After agitation the solution must be complete.

An excellent oil is also produced in Réunion and St. Mauritius, the Réunion oil having taken a foremost rank in the trade in the course of the last few years. It is distilled by a number of producers in and around St. Dénis. It is of unvarying excellence and has obtained a first-class standing.

The chief ingredient of all geranium oils is geraniol, which is isomeric with linaool and closely allied to olefinic alcohol. The various geranium oils also contain, in addition, other constituents at present unknown. It is probable that these constituents also belong to the group of esters, the presence of which is not immaterial to the odor of the oil.

In order to obtain a clearer insight into the chemical composition of geranium oils, Messrs. Schimmel & Co. of Leipsic have undertaken a careful investigation of them, the results of which are briefly subjoined.*

" In all geranium oils which have been investigated up to the present, geraniol-esters have been found, the tiglic acid-geranyl-ester usually predominating. Thus, to give an example, after saponification of 2.2 lbs. of Réunion geranium oil, an acid mixture boiling between 212° and 410° F. was isolated from the alkaline lees, which partly solidifies as tiglic acid ($C_5H_8O_2$).

The acid, recrystallized from dilute alcohol, had a melting-point of 147.2 to 149° F. Upon analysis of the silver-salt the following percentage of Ag was obtained:—

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* Report, April, 1894.

0.3146 grammes contained 0.1640 grammes Ag
0.4973 " " 0.2590 " "
Actually found: 52.13 per cent. Ag — Theory required: $C_5H_7O_2Ag$
" " 52.07 " " " — " " 52.17 p. c. Ag

Saturated with bromine in carbon disulphide, the acid was converted into the dibromide, melting at 188.6° F., as is proved by the bromine determination:—

0.3268 grammes yielded 0.4712 grammes AgBr
Actually found: Theory required: $C_5H_8Br_2O_2$
61.35 per cent. Br " " 61.54 per cent. Br

In addition to tiglic acid, butyric and valerianic acids are possibly present in the oil. From African geranuium oil also tiglic acid of 149° F. melting point was obtainable in large quantities.

A series of quantitative ester determinations of different oils shows that the percentage of esters varies in the different kinds.

Réunion oil is the richest in esters, of which it contains about 31 per cent. In all probability, when once sufficient material has been collected, the quantitative ester-determination will be found useful in ascertaining the value of an oil, in addition to the determination of its physical properties. Further data have also been brought together concerning the quantitative determination of geraniol, the most important constituent of the geranium oils.

By boiling with acetic-anhydride it is possible to convert geraniol quantitatively into geranyl acetate. This fact was ascertained by the aid of chemically pure geraniol recovered from its calcium chloride combination.

The geraniol percentage of the oil is calculated from the saponification-number of the acetylized product. The higher that number, the larger the geraniol-percentage in the sample under examination. This test may be of value (in connection with the combination of the physical properties) in estimating the quality of the oils.

The following table gives the physical data, the ester content and the geraniol content of different oils:

| Description of the samples. | Sp. gr. at 59° F. | Solubility in 3 vols. of 70 per cent. alcohol. | Opt. rot. in a 100 mm. tube. | Ester content calculated on tiglic acid geranyl ester. per cent. | Saponification number of the acetylized oil. | Geraniol content of the oil. per cent. |
|---|---|---|---|---|---|---|
| Geraniol, chemic. pure.. | 0.8835 | soluble | ±0° | — | 287.2 | 100.4 |
| Palmarosa oil ......... | 0.894 | " | —1°55′ | 15.0 | 259 | 90.5 |
| Réunion oil........... | 0.890 | " | —9°15′ | 31.3 | 241 | 84.3 |
| " ........... | 0.891 | " | —9° | 31.1 | 242 | 84.6 |
| " ........... | 0.893 | " | —8°32′ | 33.3 | 243 | 84.9 |
| " ........... | 0.891 | " | —8°8′ | 32.5 | 229 | 80 |
| " ........... | 0.891 | " | —8°57′ | 34.1 | 227 | 79.3 |
| African geranium oil ... | 0.898 | " | —8°45′ | 29.1 | 235 | 82.1 |
| Spanish " ... | 0.898 | " | —8°12′ | 23.7 | 235.5 | 85.5 |
| Schimmel & Co.'s distillate from plants grown at Leipsic .......... | 0.906 | " | —16° | 27.9 | 212 | 74.1 |

The examination of the following artificially-prepared mixtures shows the extent to which these properties vary in adulterated oils:

| | | | | | | |
|---|---|---|---|---|---|---|
| Réunion oil with 20 per cent of oil of turpentine | 0.885 | insol. | —13°28′ | 25.7 | 194 | 67.9 |
| do. with 30 per cent. of fatty oil ............ | 0.894 | " | —5°48′ | 48.2 | 234 | 81.8 |

The most noticeable circumstance is that the addition of oil of turpentine affects the solubility, but all other properties are also clearly affected. The geraniol content has been reduced to 67.9 per cent.

In the presence of fixed oils, which themselves yield a high saponification-number, the geraniol and ester percentages are no guide. In this case also the specific gravity and optical rotation undergo but little change. On the other hand, a mixture of this kind is distinguishable from normal oils by its insolubility in 70 per cent. alcohol, as well as by the fact that it only partly volatizes under steam distillation.

The geraniol estimation above referred to is based upon the supposition that the alcoholic element of geranium oil consists only of geraniol. Future investigations must show whether this be really so, or whether still other alcohols of the $C_{10}H_{18}O$ or $C_{10}H_{20}O$ formula are present.

Rose geranium oil is frequently adulterated by grass oils, further by copaiva oil, fat oils and cocoanut oil. To detect such additions shake 5 drops of the oil with 5 ccm. of 70 per cent. alcohol, whereby pure geranium oil should be dissolved. Further, grass oil is colored brown by iodine, while geranium oil is not. Nitric acid colors rose geranium oil green, andropogon oil was intensely dark yellow; sulphuric acid gives to the latter a strong odor of fat. Cocoanut oil is separated as a white substance when the adulterated oil in a test glass is for several hours cooled with ice, or a freezing mixture.

Geranium oil is used for many purposes. It serves in perfumery as a rose-like smelling substance, but is chiefly used for adulterating rose oil. It is employed as a substitute for rose oil, especially in the tobacco industry (snuff). The best commercial varieties are the French and Spanish geranium oils, the brownish colored oils being preferred. African geranium oil closely approaches the French and Spanish oils as regards quality, while, as previously stated, Réunion geranium oil now occupies a front rank in the trade. Under the name of Turkish geranium oil, a grass oil is generally brought into commerce, which, according to Gintl, is prepared from *Adropogon Pachnodes.*

### *Tropæolaceæ.*

*Indian cress oil* (*Kapuziner Kressenöl*, G). The herb and seed of the Indian cress, or garden nasturtium, *Tropæolum majus*, L., yield a volatile oil, which has been examined by A. W. Hofmann. The herb, flowers and unripe seed were distilled with steam; the distillate yielded the volatile oil to benzol; the yield was 0.025 per cent. The oil commenced to boil at 320° F., and left behind even at 572° F. quite a considerable quantity of a brown residue. The first fractions possessed an extremely offensive odor and contained sulphur, which was entirely wanting in the higher fractions. The oil proved to be nitrogenous. By rectification the boiling point of the principal mass was 438.8° F. (corrected, 449.4° F.) This oil was colorless, highly refractive, of specific gravity 1.0416 at 64.4° F., and of an aromatic odor. By heating with sodium, cyanide was developed, and by boiling with alcoholic potash lye ammonia was released. An analysis gave the formula $C_8N_7N$, and closer examination showed it to be phenylacetonitril, $CH_2C_6H_5CN$. This body could be obtained from all fractions. From the oil completely decomposed with potash lye, a small quantity of a volatile hydrocarbon could be distilled off, so that in addition to phenylacetonitril the oil seems to contain traces of a hydrocarbon and of a sulphuretted body.

### VI. *Volatile Oils of the Terebinthinæ.*

To this group belong some of the most important volatile oils, which, on account of their great fragrance, find extensive use, especially the oils of the Aurantiaceæ. The following families of plants come here under consideration: *Rutaceæ*, *Aurantiaceæ*, *Simarubaceæ*, *Burseraceæ*, *Anacardiaceæ*, and *Euphorbiaceæ*.

Most of the oils derived from the plants of the above-mentioned families contain limonene as chief constituent or by-constituent. All the oils contain also (generally in small quantity) oxygenated bodies, which appear to be the bearers of the odor. Special mention deserves the occurrence of methyl-nonyl ketone, $CH_3COC_9H_{19}$, in rue oil and perhaps in lime oil. Myrrh oil alone is heavier than water, all the rest being lighter. Most of the volatile oils of this group commence boiling at about 338° F. and are very sensitive to the action of light and air. While the oils of the *Rutaceæ* are not exactly fragrant, and those of the *Anacardiaceæ* possess a more powerful than agreeable odor, the oils of the *Aurantiaceæ* belong to the most fragrant volatile oils known, especially the costly neroli oil.

### *Rutaceæ.*

*Rue oil, oleum rutæ* (*Rautenöl*, G.; *essence de rue*, F.) All parts of the common rue, *Ruta graveolens*, contain a considerable quantity of volatile oil, which can be obtained by distillation. The leaves require to be handled with the greatest care or they provoke painful swellings. The plant is largely grown in the neighborhood of Grasse, France. One hundred and fifty to two hundred pounds of it produce one pound of oil. The oil is colorless to pale yellow, and of the agreeable but strong odor of the plant. Its specific gravity is 0.8295; it boils at 405° F., and solidifies at from 41° to 43° F. It is frequently adulterated. When pure it is miscible with glacial acetic acid in all proportions, and forms a clear solution with alcohol. Iodine dissolves in it slowly without any perceptible reaction except a darkening and slight increase of viscidity. Solution of chromate of potash has no effect upon it. If it turns reddish-brown with solution of potash and still darker with sulphuric acid, or if it fulminates with iodine, it is adulterated with some other oil.

Rue oil consists chiefly of methyl-nonyl ketone, $C_{11}H_{22}O = CH_3COC_9H_{19}$, mixed perhaps with smaller quantities of terpene and camphor, $C_{10}H_{18}O$.

*Buchu leaf oil* (*Bukkublätteröl*, G.) is obtained from various species of *Diosma*, family *Rutacæe*, shrubs indigenous to the southern portion of Africa, more particularly to the various parts of the Cape Colony. The leaves, especially those of *Diosma* (s. *Barosma*) *betulina*, yield by distillation with water 0.5 per cent. (according to others 2 per cent.) of a volatile oil possessing a peppermint-like odor. By allowing the oil to evaporate a crystalline body—diosphenol, $C_{14}H_{22}O_3$—separates out; it fuses at 181.4° F., boils at 451.4° F., and is readily soluble in alkalies. At 401° to 410° F. the oil in addition yields a fraction smelling of peppermint, which is optically inactive and has the composition $C_{10}H_{18}O$. Y. Shimoyama has recently subjected diosphenol to a closer examination, and applied to it the formula $C_{10}H_{16}O_2$. By boiling diosphenol with alcohol potash lye a new acid—diolic acid, $C_{10}H_{18}O_3 + H_2O$—was obtained.

It is solid, melts at 204.8° to 206.6° F., and dissolves with difficulty in water. By sodium amalgam the diosphenol dissolved in alcohol is converted into diolic alcohol, $C_{10}H_{18}O_2$; it forms prisms which melt at 316.4° F. and are soluble with great difficulty in alcohol and ether.

*Jaborandi leaf oil* (*Jaborandiblätteröl*, G). The leaves of *Pilocarpus pennatifolius*, or jaborandi, a shrub growing in Brazil, contain the alkaloid pilocarpine ($C_{11}H_{16}N_2O_2$), another alkaloid jaborine, and a volatile oil. The yield of oil from the leaves is 0.4 to 0.56 per cent., while the bark furnishes much less. The oil possesses an extremely powerful odor, and a mild, fruity taste. Specific gravity, 0.875; boiling point, 352.4° to 554° F. According to Hardy, the oil contains a terpene, $C_{10}H_{16}$, pilocarpene (sp. gr. = 0.852; $[\alpha]_D = +1.21$; hydrochloride, $C_{10}H_{16},2HCl$, crystalline, melting at 121.1° F.), and, above 500° F., yields distillates which solidify in the cold, the paraffine-like body thereby formed melting at 80.6° to 82.4° F. The hydrocarbon "pilocarpene" is probably limonene, this supposition being supported by the fact that Pöhl designates the fraction boiling at 345.2° F. similar to carvene, and the letter is identical with limonene (Husemann).

*Angostura bark oil* (*Angosturarindenöl*, G). By distillation angostura bark, from *Galipea Cusparia*, St. Hil., yields 1½ per cent. of oil possessing a yellow color, and a mild, aromatic taste. The oil has not yet been thoroughly examined.

### *Aurantiaceæ.*

*Orange flower oil*, *oleum aurantiorum florum*, *oleum neroli*, *oleum naphæ* (*Neroliöl*, G.; *essence du néroli ou des fleurs d'oranger*, F.) is obtained by submitting the freshly gathered flowers of either the sweet or bitter orange to the maceration or absorption-process. Other products are obtained by aqueous distillation which, though of a very agreeable odor, cannot compare with the genuine article. These are known according to their derivation as "*oil of neroli*," which is procured from the flowers of *Citrus aurantium;* "*neroli bigarade*" from the blossoms of *Citrus bigaradia* or Seville orange; and "*neroli petit grain*" from the leaves and young unripe fruit of the different species *of citrus*.

The distillation of orange flowers is effected as follows: The flowers are spread out 5 to 7 inches deep upon the stone floor of a suitable room and the calyces picked out and removed. The flowers are then brought by means of a sail-cloth funnel into a retort, and water sufficient to cover the flowers is added. The man-hole of the retort is then closed and the water brought to boiling by means of steam introduced into a coil. The water and oil distil off, and are caught in a receiver arranged like a Florentine flask. The oil rises to the surface and is retained in the receiver while the water runs off. In the receiver is a piece of wood or cork which from time to time interrupts the current of vapor from the retort. The force of the current passing down being broken by this means, the drops of oil gain time to combine and thus escape the danger of being carried along. From the Florentine flask the orange flower oil is removed by means of a pipette, filtered and brought into bottles. One thousand pounds of flowers yield about 1 lb. of oil (0.1 per cent.)

The *orange flower water* obtained in distilling the blossoms, known as *aqua naphæ*, is brought into commerce, large quantities of it being used in France and England for various purposes.

Freshly-prepared orange flower oil is almost colorless, but on exposure to the light turns red. It has a very agreeable odor of orange blossoms, and an aromatic slightly bitterish taste. The specific gravity of the genuine oil is, according to Schimmel & Co., 0.885 to 0.887. The oil has a neutral reaction, and is very sparingly soluble in water, but even the smallest possible quantity dissolved suffices to impart to the water the agreeable odor of the oil. In alcohol it is readily soluble. By mixing the clear alcoholic solution with an excess of 90 per cent. alcohol, the mixture becomes turbid, and after standing for some time a stearoptene is separated. The oil is strongly dextro-rotatory.

Although the investigations of neroli oil have not been entirely concluded, the principal mass (99 per cent.) of it may be said to consist of a terpene, $C_{10}H_{16}$ (limonene), boiling at 343.4° F., while a small quantity (1 per cent.) of a stearoptene, called aurade, constitutes the odoriferous portion, which is scarcely soluble in water.

With iodine orange flower oil effervesces and becomes strongly heated, yellow-red and violet vapors being formed; the residue is yellow-brown and soft, and of a sourish balsamic odor. Nitric acid colors the oil dark reddish-brown, and, on heating, oxidizes it vigorously. Sulphuric acid colors it dark red-brown, the acid itself becoming thereby reddish-yellow-brown; the alcoholic or aqueous solution of the oil is reddened by sulphuric acid. Warm alcoholic potash lye dissolves the oil to a clear yellow-brownish liquid. Hydrogen chloride separates from the oil a solid hydrochloride, which as regards odor, form of crystallization, and melting point, perfectly resembles that obtained from lemon oil, and has also the same composition, $C_{10}H_{16}2HCl$. Hence the hydrocarbon of neroli oil is limonene.

On account of the high price of orange-flower oil it is largely adulterated, especially with alcohol, castor oil, petit grain oil, bergamot oil, copaiva oil, etc. Mierzinski even states that no genuine orange-flower oil is brought into commerce, the usual commercial article consisting of a mixture of about four-eighths orange-flower oil, one-eighth bergamot oil, and three-eighths petit grain oil. Sophistication with oils of other varieties of citrus may be detected as follows: Dissolve a few drops of the suspected oil in about 50 drops of 90 per cent. alcohol and add, with constant shaking, 15 to 18 drops of sulphuric acid. Pure oil acquires a reddish-brown color, and the quicker this takes place, the fresher the oil. Adulterated oil gives either a pure red or reddish-yellow liquid. Fat oils and copaiva oil in orange flower oil may, according to Schramm, be detected by soaking a wick with an alcoholic solution of the oil and igniting. When the alcohol is burned an odor of the admixed fat oil or of copaiva oil is evolved.

Orange flower oil is chiefly used for fine perfumeries.

Under the name "nerolin" a product possessing the odor of fresh orange flowers in concentrated form is brought into commerce by Schimmel & Co. It is recommended for perfuming purposes, especially of soaps. It is a white, crystalline, absolutely dry body, which dissolves in 30 parts of 95 per cent. alcohol or in 25 parts of fat oil, but scarcely in water.

*Orange peel oil, oleum aurantii corticis,* (*Pomeranzenschalenöl,* G.; *essence d' écorce d' oranges,* F.) is obtained from the peels of both the bitter and sweet orange in a manner similar to that of lemons (which see). The yield is 2 to 2.7 per cent. The best quality of orange-peel oil comes from Calabria; an inferior quality from Messina.

Orange-peel oil from sweet oranges is yellow to brown, but by rectification becomes clear as water. It has a mild, spicy taste, and an agreeable orange odor. The oil prepared from ripe fruit has, in a crude state, a specific gravity of 0.8508 at 68° F., and in a rectified state, of 0.842 at 68° F., while crude oil prepared from nearly ripe fruit has, according to Hofmann, Pinther and Soltsien, a specific gravity of 0.8451 at 68° F. The boiling point lies between 345.2° and 356° F. With equal volumes of 95 per cent. alcohol the oil gives a turbid solution, which, however, gradually becomes clear. The odor of the oil is changed by distillation by itself or with water, and also becomes rancid when the oil is badly kept. The oil, however, possesses a considerable constancy of odor. It gives a very slight chlorophyl spectrum.

Orange-peel oil from bitter oranges is, like that from sweet oranges, limpid and yellowish, but when carelessly kept, becomes thickly-fluid and darker. It possesses a powerful, spicy, refreshing odor and an aromatic bitterish taste, which differs, however, from sweet orange peel oil. Its specific gravity lies between 0.83 and 0.86. It dissolves clear in absolute alcohol, but not in 5 parts of 90 per cent. alcohol.

Orange-peel oil consists almost exclusively of limonene and contains, in addition, only about 0.3 per cent. of myristicol and

2.8 per cent. of a body, $C_{20}H_{30}O_{3}$. The latter has not been closely examined and probably is formed by oxidation from the oil.

Iodine is vigorously absorbed by orange-peel oil, it taking up, according to Williams, 342 to 348 per cent., the oil separating thereby a dark-colored iodine combination. The absorption of caustic potash is small (0.38 to 0.39 per cent.). In alcoholic potash lye the oil dissolves clear only with the assistance of heat, but on cooling mostly separates out with a golden-yellow color.

Orange-peel oil is particularly adulterated with alcohol, oil of turpentine and inferior oil of other species of *Aurantiaceæ*. Orange-peel oil is used in the manufacture of liqueurs and perfumery, and for the adulteration of orange-flower oil. In medicine it is almost exclusively employed as a flavoring or perfuming ingredient of medical compounds.

*Mandarin oil.* The mandarin orange tree is a variety of the orange tree, according to Luca, *Citrus Bigaradia Simensis* and *Citrus Bigaradia myrtifolia.* By expressing the peels a golden yellow, clear, very mobile oil of an agreeable taste and an odor reminding one of orange peel is obtained. At 352.4° F. the oil passes over almost completely, only a small residue, the coloring matter, remaining behind. The distillate is colorless, tastes and smells like the oil, and possesses a specific gravity of 0.852 at 50° F. Its composition is that of a terpene, $C_{10}H_{16}$. The oil is insoluble in water, imparting, however, its odor to it. It is soluble in 10 times its volume of alcohol and in every proportion in carbon disulphide, ether or glacial acetic acid. It dissolves iodine, bromine, resins, oils, wax, phosphorus, sulphur, etc. By concentrated sulphuric acid the oil is colored red, the color becoming yellow by the addition of water and the mixture turbid. By the application of heat the oil is carbonized by sulphuric acid. Cold nitric acid colors the oil yellow, warm nitric acid resinifies it. When allowed to stand for several days with concentrated hydrochloric acid, a crystallized dihydrochloride, $C_{10}H_{16},2HCl$, consisting of small transparent laminæ, is formed. The oil is strongly dextro-rotatory and in a pure state fluoresces bluish. The terpene of mandarin oil is probably limonene.

*Petit grain oil* (*Petitgrainöl*, G.; *essence de petit grain*, *de néroli petit grain*, F.). As previously stated, this oil is obtained by distilling the unripe fruits, flowers, leaves, young shoots, etc., of different species of citrus. The best quality of oil is prepared from the leaves of *Citrus Bigaradia*, Duham. The shoots are collected, tied up in bundles, and delivered to the factories. The stouter shoots are crushed, the leaves and young branches cut up, and the mass together with water is brought into the still. Commercial petit grain oil, however, is generally a mixture of oils of various derivations.

The smell of petit grain oil, while resembling that of orange flowers, is by no means so fine. Its color is yellowish, and its taste similar to that of orange-peel oil and quite mild. Its other properties vary very much according to the raw material used. Gladstone gives its specific gravity as 0.8765 at 60° F. It is dextro-rotatory, and soluble in an equal volume of the strongest alcohol. Its composition is not accurately known, but limonene is no doubt the principal constituent. Besides the French petit grain oil, which is highly valued, a product is brought into commerce from Paraguay, which has become a strong competitor with the French and Algerian oils.

Petit grain oil is chiefly used for perfuming soaps and in the preparation of perfumes, especially of Cologne water. It is adulterated in a similar manner as orange-flower oil, but is also used largely for the adulteration of finer qualities of citrus oils.

*Bergamot oil*, *oleum bergamottæ* (*Bergamotöl*, G.; *essence de bergamote*, F.). The rind of the bergamot orange, *Citrus Bergamia*, Risso; *Citrus Bergamia*, var. *vulgaris*, Risso et Poiteau, yields a volatile oil of delicious odor. The rind is grated, and the oil running off, separated from the aqueous fluid and cellular substance by means of a separatory funnel, or the grated mass is distilled in a current of carbonic acid.

The rind of 100 bergamot oranges will yield about three ounces of oil. It is of a pale green or yellowish color, becoming, however, darker by age. In storing oil of bergamot great care must be exercised to exclude light and air, as it is one of the most changeable oils, and soon acquires an odor resembling that of turpentine. The specific gravity ranges from 0.856 to 0.888 at 59° F., and the boiling points from 365° to 397.5° F. It solidifies at 11° F. It is readily soluble in alcohol, and is, therefore, very frequently adulterated with it, and also with oil of orange or lemon. In the presence of the oil of orange the alcoholic solution is turbid. Iodine exerts a very energetic effect upon the oil, violet vapors being formed. Four parts by weight of pure oil of bergamot, 3 parts by weight of alcohol, and 1 part by weight of nitric acid, give in a short time a solid crystallized body. Sodium amalgam has no effect upon oil of bergamot.

Oil of bergamot consists mainly of limonene mixed with a small quantity of dipentene and bergaptene, as well as not inconsiderable quantities of a body not yet thoroughly known.

Oil of bergamot is adulterated with alcohol, almond oil, and orange-peel oil, but chiefly with lemon oil.

An addition of alcohol may be detected by an addition of equal quantities of pure oils of olive and almond. In the presence of alcohol the oils do not mix, which is, however, readily the case with pure oil. Certain adulterations may also be established by the addition of alcohol; pure oil becomes pale gray-yellow, forms a sediment which is flaky and adheres firmly, and in two days becomes quite inconsiderable.

Pure bergamot oil should give a clear solution with ½ part or more of alcohol of 90 per cent. This test, however, does not appear to hold good in all cases, and in order to guard against adulterations with fatty oils it is advisable in all cases in which bergamot oil fails to give a clear solution with 1½ to 2 volumes of 80 per cent. alcohol to test the residue of evaporation. This is done by weighing from 2 to 5 grammes of oil of bergamot in a glass dish of known weight and heating it on a water bath until it has become quite odorless. The residue should not exceed 6 per cent. of the weight of the sample. As the whole of the fatty oil is present in the residue, the quantity of foreign fatty substances which may have been added to the sample can be readily ascertained by deducting 6 from the total percentage formed.

Bergamot oil is chiefly used in perfumery for the preparation of cosmetics, perfuming of soaps, and also in the preparation of liqueurs. Mixed with other volatile oils it increases their fragrance, and imparts, especially to spice oils, a delicacy like no other addition.

*Lime oil or limetta oil, oleum limettæ* (*Limettöl*, G.; *essence de lime ou limette*) is derived from the rind of the fruit of *Citrus Limetta*, Risso. The oil is obtained in the same manner as lemon oil, which it somewhat resembles. Its mean specific gravity is 0.8734 at 84° F. The oil made by the écuelle process is of a decidedly yellow color, varying in intensity, being darker in the fresh product. The difference in flavor and aroma is so marked as scarcely to require any other means of distinguishing the oil made by the écuelle process from distilled oil, the first having a decided and fragrant lemon smell, whilst the distilled oil is very inferior, frequently possessing little more than the smell of turpentine. Large quantities of lime oil are manufactured in Dominica. While the lime (*Citrus Limetta*, Risso) growing in Southern Europe has a sweet juice, that of the West Indian fruit is acid. According to the latest investigations the West Indian tree is to be regarded as a variety of *Citrus medica*, which has been named *C. medica var. acida*, Brandis. It is cultivated chiefly in Montserrat, Dominica, and Jamaica for the sake of the fruit-juice and the essential oil. The former is expressed by crushing the fruit between heavy rollers; it is exported either in the original condition, or it is first evaporated to about $\frac{1}{10}$ its volume. During this evaporation an essential oil is obtained as a by-product and put upon the market as "oil of limes." It has an odor not particularly pleasant nor like that of citral, and is quite different from the expressed oil. The latter is generally obtained by the écuelle process. It has a very agreeable and strong odor of citral. In commerce it is known as limetta oil. Several oils investigated by Schimmel & Co.* showed the following constants: *Distilled oils from Dominica:* No. 1. Specific gravity, 0.868; optical rotation, +38°35′; boiling point, between 347° and 392° F. No. 2. Specific gravity, 0.867. *Expressed oils:* No. 1, from Montserrat. Specific gravity, 0.882; optical rotation, +35°40′. No. 2, from Dominica. Specific gravity, 0.882; optical rotation, +37°55′.

Mierzinski gives the following as the constituents of lime oil: Limonene $C_{10}H_{16}$, boiling point 348.8° F.; terpinol $C_{10}H_{17}OH$; methyl-nonyl ketone $CH_3COC_9H_{19}$; diterpene $C_{20}H_{32}$. According to W. A. Tilden and Ch. R. Beck, from the oil of *Citrus Limetta* obtained by the écuelle process a substance of the formula $C_{16}H_{14}O_6$ crystallizes out, which they call limettin. It forms small yellow needles with a fusing point of 249.8° to 251.6° F., and in melting yields with potassium, phloroglucinic, acetic and formic acids. Oils of lemon and bergamot yield similar substances.

*Lemon oil, oleum limonis, oleum citri* (*Citronenöl*, G.; *essence de citron, de limon*, F.) is obtained by various processes from the rinds of lemons. The best and most delicately scented oil is obtained by the so-called sponge process in use in Southern Italy and Sicily. The rinds are soaked from fifteen to twenty-five minutes in water, to which sometimes a little soda is added. They are taken up singly in the right hand, and the outer surface of each is firmly pressed against a large and rather hard-grained sponge held in the left hand and secured by a strap. Two or three sharp turns of the wrist impart what might be called a screw pressure to the rind, thus effectually fracturing the oil cells, the sponge absorbing the contents. The sponge is constantly held over an earthen jar and occasionally squeezed into it. The fluid in the jar quickly separates into three different products—the dregs or deposit of mucilaginous and cellular matter, some fruit juice, and the pure oil which floats on the top. The latter, when bright and clear, is passed by means of a small glass siphon into the cans of thin copper in which, after sealing, it is stored away for export. For 1 lb. of such hand-pressed oil or *essence préparée à l'éponge*, 500 to 750 fruits are required.

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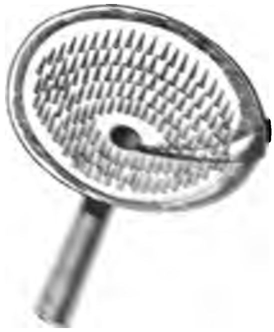
* Report, October, 1894.

Another method is that of the *écuelle*, an instrument much used in Southern France. It consists, Fig. 297, of a round shallow pan, generally of copper, having a receptacle for the oil at its lowest part and a lip on one side for pouring, and studded on its concavity by strong, blunt spikes. The workman takes the fruit and rolls it gently, but quickly, around the inside of the écuelle; the spikes prick the oil sacs, whereupon the oil, running down the spikes and the concavity of the pan, collects in the reservoir at the lowest part. The oil is filtered and then poured into clean glass bottles in which the impurities are allowed to settle. This appliance gives the *essence à l'écuelle or essence au zeste.*

FIG. 297.

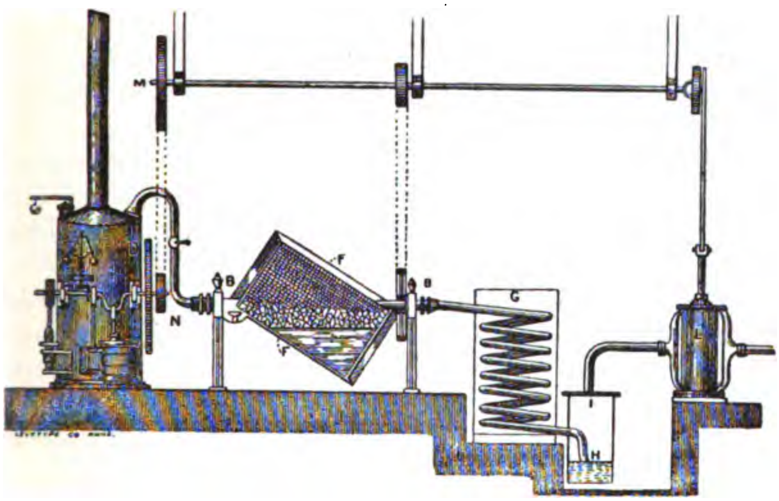


The third method of obtaining the oil is by expression. The grated rind is placed in hair mats and subjected to powerful pressure by means of a screw or lever press.

The process of distillation is carried on as follows: The peels, which should be from select fruit, are sprinkled with powdered salt and a few hours afterwards sufficient water to moisten them is poured over them. A day or two afterwards more water is added, and the whole is distilled until either no more oil separates, or the steam, at first purely fragrant, begins to acquire a rank and rather unpleasant smell. The oil obtained by distillation is inferior to the others.

Lately a combination of the écuelle and distilling process has been introduced by Domenico Monfalcone, which has given excellent results. It is shown in Fig. 298. *A* is a hollow cylinder of sheet iron, the interior surface of which is studded with a large number of small metallic knife points. This cylinder revolves on two axles attached to it at diagonally opposite points. The cylinder having been half charged with lemons, together with a small quantity of water, is set in motion by aid of the shafting and pulleys driven by the small steam-engine. The fruits are thereby made to come in contact with the metallic lancets, and their whole surface gradually becomes punctured sufficiently to rupture the cells containing the volatile oil, which escapes. The cylinder *A* has double walls, the space between them being intended for the circulation of a current of steam which is admitted to heat the water and to facilitate the extraction of the oil, while at the same time the vapors are rarefied or aspirated by the vacuum pump *L*. The axles of the cylinder on both sides are hollow; that on the side looking towards the engine is imbedded in such a manner that steam from the boiler *D* may be admitted at will, either into the double walls of the cylinder or into the interior of the cylinder itself, while the hollow passage in the other axle communicates with a condensing worm *G*, the outlet of which, *H*, descends

FIG. 298.



into a cylindrical vessel *I*, intended to receive the condensed products, consisting of water and essential oil. When the apparatus is first set in motion the pump *L* begins to produce a vacuum during the first revolutions of the cylinder. Steam being now cautiously admitted into the double walls of the cylinder, the water is raised to boiling at a comparatively low temperature, and the vapors charged with the essential oil rapidly pass over into the receptacle, where they separate into two layers. The product obtained by this process is claimed to be equal in quality to that obtained by mechanical means, and the yield nearly double.

During the months of November to March the average yield from 1,000 lemons in the factories of Palermo is 320 grammes of oil; in those of Messina, where a better quality of fruit is employed, the yield is about 400 grammes. The same number of lemons yield about 10 gallons of acid juice, the collection of which is, of course, a necessary accompaniment of the manufacture of volatile oil.

Expressed lemon oil is of a yellowish color, possesses a very agreeable lemon odor and taste, the latter being at the same time very aromatic. It has an acid reaction, and a specific gravity of 0.855 to 0.859 at 59° F. Crude lemon oil boils between 338° and 348.8° F., and rectified oil at 328° F. The rotatory power of crude oil is from + 56° 30′ to + 60° 32′, and that of rectified oil + 60° 20′ to 64° 30′. By heating in a closed tube to 572° F., the rotatory power of lemon oil is not changed, while with the same treatment that of oil of turpentine decreases materially. As oil of turpentine is notoriously almost the only adulterant of lemon oil, of which it lowers the specific rotatory power considerably, while on the other hand increasing its specific gravity, the determination of these two points becomes of supreme importance.

In the following tables the figures obtained by recent investigations of Schimmel & Co.* are summarized:

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* Report, April, 1893.

## *Lemon Oils of Normal Characteristics.*

| Origin of the Sample. | Spec. rotatory power (100 mm). | Spec. gravity at 15° C. |
|---|---|---|
| Schimmel & Co.'s pressing | + 61° | 0.858 |
| Reggio S. | + 62° | 0.858 |
| “ “ | + 60° 50′ | 0.859 |
| “ “ | + 61° 40′ | 0.858 |
| Messina A | + 60° 30′ | 0.859 |
| “ “ | + 60° 20′ | 0.859 |
| “ “ | + 62° | 0.858 |
| “ “ | + 60° 20′ | 0.859 |
| “ J | + 61° | 0.858 |
| “ “ | + 61° 20′ | 0.858 |
| “ “ | + 60° 20′ | 0.858 |
| “ “ | + 61° 40′ | 0.859 |
| Catania R | + 63° 40′ | 0.858 |
| “ “ | + 62° | 0.858 |
| “ “ | + 63° 20′ | 0.858 |
| “ “ | + 63° 20′ | 0.858 |
| “ “ | + 64° 30′ | 0.858 |
| Messina B | + 64° 20′ | 0.858 |
| Palermo L | + 62° 20′ | 0.858 |
| Messina B | + 62° | 0.859 |
| “ “ | + 61° | 0.859 |
| “ P | + 66° 40′ | 0.857 |
| “ GR | + 62° 40′ | 0.858 |
| “ K | + 62° 20′ | 0.859 |
| ***Suspected Lemon Oils.*** | | |
| Reggio S | + 59° | 0.859 |
| Messina A | + 59° | 0.859 |
| “ “ | + 57° | 0.859 |
| “ “ | + 57° | 0.859 |
| “ “ | + 55° 40′ | 0.859 |
| “ B | + 58° 40′ | 0.859 |
| “ H | + 55° | 0.860 |
| ***Adulterated Lemon Oils.*** | | |
| Messina B | + 52° | 0.860 |
| “ P | + 44° | 0.861 |
| “ K | + 31° | 0.865 |
| “ GA | + 23° 20′ | 0.865 |
| Leipsic WS | + 16° 42′ | 0.873 |
| “ “ | + 34° | 0.835 |

The oil last mentioned contained 30 per cent. of alcohol and had been colored with a yellow dye.

The above-mentioned figures prove that all good commercial oils agree in their characteristics with that pressed by Schimmel & Co. from fresh lemons, whereas all the adulterated oils, without exception, show a higher specific gravity and a smaller rotatory power. These contain, therefore, a proportionally larger or smaller addition of turpentine.

Under these circumstances the following tests should be required for good, pure Lemon Oil:—

Rotatory power, 100 mm column, not below + 60°.
Sp. gr. at 15° C. from 0.858 to 0.859.

If these tests are applied with due care, adulteration by turpentine oil cannot pass undetected.

So far as known the chief constituent of lemon oil is *limonene*, $C_{10}H_{16}$; it contains also small quantities of pinene, according to Lafont, about 1.67 per cent. Further there are present one or several oxygenated bodies, of which nothing certain is known; this body is perhaps cineol, $C_{10}H_{18}O$, with which, however, the boiling points found do not agree. Many lemon oils appear to contain also a small quantity of cymol, $C_{10}H_{14}$.

According to Schimmel & Co.* lemon oil contains 7.5 per cent. of an aldehyde $C_{16}H_{16}O$ called *citral*, of specific gravity 0.899 at 59° F., and boiling point between 442.4° and 444.2° F. It has the odor of lemon oil and dissolves clear in alcohol.

Lemon oil, as previously mentioned, is chiefly adulterated with oil of turpentine and alcohol.

Lemon oil is largely used in the perfumery industry, it forming, for instance, a constituent of Cologne water. This may readily be detected by the pronounced lemon odor which the perfume shows on adding a few drops of ammonia. Mixed with oils of rosemary, clove and caraway, it yields a perfume of a very agreeable odor. For compound odors an alcoholic solution of lemon oil (5.29 to 7 ozs. of oil to 5 quarts of alcohol according to Piesse; 7 to 8.8 ozs. of oil to 4 quarts of alcohol, according to Hirzel) is usually employed. However, lemon oil should not be used for perfuming fats, since the danger of their becoming rancid is promoted by its great tendency to oxidation. Lemon oil is also occasionally employed in medicine.

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*Reports 1888, 1890.

*Lemon oil free from terpene.* The manufacture of a concentrated, *i. e.* wholly or partly terpene-free lemon oil is now carried on in Messina. According to E. Geisler and Schweissinger it has a specific gravity of 0.8981 to 0.9003 at 59° F., and boils at 419° to 455° F. It is colorless, and has a more intense taste and odor than ordinary lemon oil. It contains considerably more oxygen than ordinary lemon oil, and dissolves with great ease in 88 per cent. alcohol.

*Lemon-flower oil.* From the flowers of the lemon tree is obtained, according to Piesse, a very fragrant oil, which is used especially in the preparation of Hungary water. It oxidizes very readily.

*Artificial lemon oil.* H. Deville found that by treating the hydrate of turpentine oil with hydrochloric acid a dihydrochloride is obtained which melts at 111.2° F., and by treating at as low a temperature as possible with potassium, yields a terpene of the agreeable odor of lemon. More recently, Bouchardat and Lafont have obtained from French turpentine oil an artificial lemon oil by dissolving the portion boiling at 311° to 314.6° F. in an equal weight of glacial acetic acid, treating the solution at below 104° F. with chromic anhydride and distilling off from the product of reaction, the fraction boiling at 345.2° to 352.4° F. They call this fraction levo-rotatory terpilene, but could not free it from an admixture of about 16 per cent. cymol. In its properties and odor the fraction closely resembles lemon oil.

*Citron oil or cedrat oil, oleum de cedro* (*Cedro- or Cedratöl*, G.; *essence de cédrat*, F.). The rugged and thick rinds of *Citrus medica*, Risso, constitute the candied citron of commerce. However, a volatile oil may also be obtained from them by the ordinary methods. The oil is very limpid, slightly yellowish, and possesses a strong, extremely fine odor of lemon, and an aromatic taste. Its specific gravity ranges from 0.8517 to 0.869. It slightly reddens litmus paper and remains liquid at —4° F., but when carelessly kept becomes thickly fluid. It is used for the same purposes as lemon oil. Cedrat essence is obtained by dissolving 1¾ ozs. of cedrat oil in 5 quarts of alcohol, 8½ drachms of bergamot oil being occasionally added (Piesse).

*Sweet lemon oil* is obtained from the rinds of *Citrus Lumia*, Risso. The oil is obtained especially in Squillace, Calabria, by expressing the rinds of the fruit which in that locality is called Limi di Spagna. The oil is dark yellow; after distillation water-clear. It commences to distil between 266° and 356° F.; the principal portion, however, passes over between 356° and 374° F. At 392° to 428° F. white empyreumatic vapors are evolved, while a dark-brown body remains in the residue. From the principal fraction (356° to 374° F.) a terpene, $C_{10}H_{16}$, may be isolated, which has a specific gravity of 0.853 at 64.4° F., is insoluble in water, sparingly soluble in alcohol, readily in carbon disulphide and ether, and yields solid as well as liquid hydrochlorides. The solid hydrochloride has the formula $C_{10}H_{16},2HCl$, melts by moderate heating, and possesses a peculiar odor. The terpene is dextro-rotatory. The properties mentioned above indicate that limonene is the principal constituent of cedrat oil.

### *Polygalaceæ.*

*Senega oil* (*Senegawurzelöl*, G.) Senega, *Polygala Senega*, L., is indigenous to North America from Canada to South Carolina and westward to Wisconsin. The root contains fat oil, resin, and volatile oil, as well as 2 to 4 per cent. saponin, a yellow coloring matter, and malates. However, only the fresh root contains volatile oil, about 0.33 per cent., which is interesting on account of its consisting of valerianic acid ester and methyl salicylate. White senega root from *Polygala alba* contains only traces of methyl salicylate, and Japanese senega root from *Polygala tenuifolia* none; the latter smells of patchouli.

### *Simarubaceæ.*

*Evodia oil* (*Evodiaöl*, G.). From the fruit of *Xanthoxylum Hamiltonianum*, indigenous to Java, an oil is obtained which has a pale yellow color, a specific gravity of 0.84, and possesses an agreeable odor of oils of geranium and bergamot. The oil has been recommended by H. Helbing for deodorizing iodoform.

From the fruit of *X. piperitum*, D. C., is obtained the *Japanese pepper oil.* Yield 3.16 per cent., odor like lemon. Specific gravity, 0.973; boiling point between 320° and 437° F.

### *Burseraceæ.*

*Myrrh oil, oleum myrrhæ* (*Myrrhenöl*, G.; *essence de myrrhe*, F.). The gum resin known in commerce under the name of myrrh is obtained from *Balsamea myrrha*, Engl., = *Balsamodendron Myrrha*, Nees. It forms irregular, yellow, reddish or red-brown, transparent, brittle pieces of balsamic odor and aromatic, bitter taste. Genuine myrrh contains, according to Braconnot and Brandes, 23 to 27.8 per cent. resins, 2.5 to 2.6 per cent. volatile oil, 12 to 9.3 per cent. mucilaginous matter, and 0 to 3 per cent. of other constituents (salts, impurities) while East Indian myrrh (*Myrrha indica s. nova*) contains, according to Ruickholdt, 44.76 per cent. resin, 2.183 per cent. volatile oil, 40.818 per cent. gum, 1.475 per cent. water and 7.65 per cent. other constituents. According to the latter author, the volatile oil is obtained by extracting the myrrh with alcohol, expelling the latter and distilling the residue with water. He obtained 2.2 per cent. of the weight of myrrh oil. It is, however, more convenient to distil the myrrh itself with 4 parts, by weight, of water until empyreumatic portions commence to distil over. The yield is said to amount to 4.4 to 5 per cent. Myrrh oil is colorless, but becomes yellow by age. It is limpid, but by exposure to the air becomes thickly-fluid, smells of myrrh and has a taste at first agreeable and then balsamic, camphor-like. It dissolves in 2 or 3 parts alcohol, readily in ether and glacial acetic acid, and forms a turbid mixture with water. Specific gravity 1.0819 at 45.5° F. It is levo-rotatory, and commences to boil at 510.8° F.

O. Köhler gives as chief constituent of myrrh oil a body, $C_{10}H_{14}O$, but whether it is carvol, thymol, or isomeric with them, is not yet decided.

By shaking the ethereal solution of myrrh oil with hydrochloric acid, a lower dark purple-red layer containing the oil is formed. A mixture of the oil with sulphuric acid acquires a dark red color, and one with nitric acid a cloudy, pale red color. By dissolving the oil in carbon disulphide and introducing bromine, the solution acquires a deep violet color.

Medicinally myrrh oil is frequently used as a substitute for myrrh. In perfumery it is used in the preparation of fumigating pastilles, etc.

*Olibanum oil, oleum olibani* (*Weihrauchöl*, G.; *essence d'oliban ou d'encens*, F.). The genuine olibanum or frankincense is derived from East Indian species of *Boswellia*, especially from *Boswellia Carteri*, Burtw., or *B. serrata*, Stckh. This tree grows in Hindostan and on the Coromandel coast. African or Arabian olibanum is derived from *Boswellia papyrifera*, Hochst.

Olibanum or frankincense forms pale yellow, more rarely reddish, transparent or opaque pieces, which are brittle and show a splintery fracture, possess a slightly balsamic odor and a bitter and pungent taste. By distillation with water frankincense yields about 4 to 5 per cent. of volatile oil, the principal mass passing over in the commencement of distillation.

Olibanum oil is transparent and colorless, and has a pleasant turpentine-like odor. It has a specific gravity of 0.866 at 78.8° F., and of 0.872 at 75.2° F. It boils between 320° and 338° F. It is soluble in all proportions in ether and absolute alcohol. It is slightly levo-rotatory. As far as known olibanum oil is composed chiefly of levo-pinene, perhaps a small quantity of dipentene, and certainly an unknown oxygenated body. Olibanum oil is resinified by caustic alkalies; with iodine it forms a thick, red mass, heat being developed. By sulphuric acid it is colored red. In regard to its reaction it resembles elemi oil.

*Elemi oil, oleum elemi* (*Elemiöl*, G.; *essence d'elemi*, F.), The botanical source is undetermined. West Indian elemi is said to be derived from *Amyrus Plumieri*, D. C., while others ascribe it to *Isica Icicariba*, D. C., and *Canarium commune*, L., the latter tree being indigenous to the Philippine Islands. All the trees mentioned belong to the family *Burseraceæ*. The resin is yellow or greenish-yellow, mostly soft, of a fennel-like odor, and balsamic bitter taste. The West Indian variety is the best quality; while Brazilian, Mexican, East Indian and African elemi are of less value and seldom occur in commerce. For obtaining volatile oil Manilla resin also is frequently employed. The volatile oil is obtained by distilling the resin with water. The yield is considerable, amounting on a small scale to 10 per cent., and on a large scale to 17.6 per cent. (from Manilla resin).

Elemi oil is water-clear and limpid. It possesses an odor of fennel, mace and dill, and has a pungent, aromatic taste. Specific gravity 0.87–0.90 at 59° F.; boiling point between 330.8° and 348.8° F., the principal mass boiling between 341.6° and 348.8° F. In alcohol as well as in ether the oil is readily soluble. It is strongly levo-rotatory.

Elemi oil consists chiefly of dipentene and phellandrene. In the oil examined by Wallach, the latter was found to be dextro-phellandrene, while Deville found levo-phellandrene. In addition the oil contains smaller quantities of polyterpenes and oxygenated bodies and traces of amyrin.

With iodine the oil becomes strongly heated, and is converted into a thick red mass. By cold nitric acid it is colored brown yellow and by hot nitric acid it is resinified, very violent reaction taking place thereby. At the ordinary temperature sulphuric acid colors the oil red and, on heating, carbonizes it. Elemi oil is largely used for perfuming soap.

*Linaloe oil and Licari oil.* The statements regarding the derivation of linaloe oil are extremely contradictory, some considering linaloe oil and licari oil as two different kinds of oil, while others regard them as identical.

According to Roscoe, by the aloe referred to in the Bible is understood the wood of *Aquilaria Agallochum*, Roxb. (family of *Aquilarineæ*), a tree indigenous to Hindostan and neighboring islands. This aloe wood (*Lignum Aloes*, which has been corrupted into linaloe) is at present used as perfume only in the East Indies. On the other hand, since the 17th century Mexico has furnished a fragrant wood known as aloe wood. The linaloe wood sold at the present time is derived from *Icica altissima* (family *Burseraceæ*). Another species of *Icica* (*Icica Icicariba*, D. C.) yields, according to Seubert, the American anime. According to Mierzinski, linaloe oil is derived from another Mexican species of the family *Burseraceæ*, viz., *Elaphrium graveolens*, Knuth, the Mexican lemon tree. Piesse also is of the opinion that linaloe oil is obtained from the Mexican lemon tree. However, in connection with this it deserves mention that, according to Seubert, under "lemon wood" or "West Indian rose wood" is also understood the wood of *Amyris balsamifera*, L. The species Amyris is classed by some among the *Terebinthaceæ* and by others among the closely allied *Burseraceæ*. Another authority considers *Amyris Linaloë* as the source of linaloe oil; it is said to grow in the hot valleys of the western Cordilleras. Finally Gladstone mentions as aloe wood a material which might perhaps be derived from *Aloëxylon Agallochum* of the family *Caesalpiniaceæ*.

While many consider linaloe and licari oils identical, Mierzinski and Piesse are of the opinion that licari oil is derived from the wood of the white cedar, *Licaria canali* or *Acrodiclidium*, indigenous to French Guiana, which is known in commerce as *bois de rose femelle*. According to Piesse the plant belongs to the *Laurineæ*. However, according to Seubert, the white cedar wood is derived from *Cypressus thujoides*, L., and from *Taxodium distichum*, Rich., two trees indigenous to North America and Mexico.

In order to ascertain whether the linaloe oils from the two kinds of wood, from Cayenne (French Guiana) and Mexico met with in commerce are practically and scientifically identical, Messrs. Schimmel & Co. obtained several hundredweights of each kind of wood direct from the producing countries. Following are the results of their examination: *

"Even in their external characteristics the two kinds of wood are so different that a common botanical origin is scarcely conceivable. The wood from *Cayenne* has a smooth bark of quite uniform thickness all round the wood. In section it exhibits a smooth surface on which the annual rings with their slightly different shades of color are distinctly visible. This wood is specifically heavier than the Mexican, and its odor is less intense. *Licaria guianensis*, Aubl. (*Lauraceæ*), is believed to be its source. In commerce the wood, as previously mentioned, is called *bois de rose femelle* (female rose-wood).

"The material imported from *Mexico* has a corky bark marked by well-defined furrows running in a vertical direction. In section it exhibits a bright orange-yellow color at the margin, becoming brownish towards the middle. Instead of the regular rings it has quite irregular marks, something like flower-diagrams, which stand up plainly from the ground-color of the wood, owing to their brownish lines. The wood is less homogeneous in structure than that of Cayenne, but more porous and spongy. Its botanical origin does not seem to have been yet determined, but it is believed to be produced by a species of *Amyris*. In Mexico it is also known as *bois de citron* (lemon-wood) or West Indian rose-wood."

*Linaloe oil.* The oil is a colorless liquid of an agreeable odor reminding one of lemon oil, so that a mixture of the oil with a little rose oil may pass as an excellent imitation of the oil from citrus flowers. The oil is quite constant in the air (Piesse). According to Gladstone, its specific gravity is 0.8702 at 64.4° F., and the greater portion of the oil passes over at 392° F. This distillate has a specific gravity of 0.864, and probably the formula $C_{10}H_{18}O$.

*Licari oil* possesses, according to Piesse, an odor reminding one at the same time of roses and lemon, and resembling that of linaloe oil. It forms an almost colorless liquid specifically

* Report, October 1894.

lighter than water. It generally contains some water, this being the reason why the oil becomes turbid at 4° F., microscopic needles of ice being formed. To obtain the oil pure it is allowed to remain in contact with calcium chloride for some time, and is then distilled off. This distillate boils at 388.4° F., and has a specific gravity of 0.868 at 59° F. The oil is levo-rotatory, soluble in ether and glycerin, and is not changed by potassium. Its composition corresponds to the formula $C_{10}H_{18}O$ (Morin).

Schimmel & Co. have repeatedly distilled the Cayenne wood and ascertained the following constants for the oil:

1892. Specific gravity 0.873. Optical rotation —16°.

1893. Specific gravity 0.877. Optical rotation —15° 7′.

Both products are soluble in 2 parts of 70 per cent. alcohol.

According to Schimmel & Co. the linaloe oil of commerce is almost exclusively of Mexican origin.

The distillate from the Mexican wood varies considerably from the above. The figures it yields are as follows:

Specific gravity 0.898; optical rotation —7° 33′. Soluble in 2 parts of 70 per cent. alcohol. The commercial article imported from Mexico possesses uniformly a somewhat lower specific gravity, probably due to the primitive mode of preparation.

Linaloe oil is subjected to considerable sophistication, especially with cocoanut oil, one sample having been adulterated to the extent of 20 per cent. Placed in a freezing mixture, the oil solidified to a butter-like mass and, on addition of alcohol, a white sediment remained visible even at the ordinary temperature. This test may be recommended in all cases. A further valuable indication is given by the solubility test. One part of linaloe oil should form a clear solution with 2 parts of 70 per cent. alcohol.

Barbier and Bouveault have given some notes on the oxidation products of licareol (linalool). Gentle oxidation with chromic-acid mixture gave rise to acetone, "licareal" $C_{10}H_{16}O$, methylheptenone, acetic, formic and methylheptenone-carbonic acids ($C_8H_{13}OCOOH$); energetic oxidation yielded terebinic acid as well as acetic and formic acids.

The authors believed "licareal," which, as they themselves admit, was not quite pure, to be different from citral (geranial) because it gave with paramidophenol a compound melting at 254.3° F. while the corresponding compound of citral melts at 240.8° F. To this question the following comments may be contributed.

The oxidation products of linalool were investigated some time ago in the laboratory of Schimmel & Co. when an aldehyde $C_{10}H_{16}O$ was obtained which, on account of its physical properties and its chemical behavior, was declared identical with the citral discovered in the same laboratory in 1888. This observation was subsequently confirmed by Tiemann and Semmler.

The detection of citral with ease and certainty has been recently made possible by Doebner's reaction. Schimmel & Co. have availed themselves of this reaction to decide the question as to the identity of citral from various sources, and are able to affirm that both the natural aldehyde of lemon and of lemongrass oils and the citrals obtained by oxidation of linalool (licareol) and of geraniol (lemonol, rhodinol, etc.) yielded with the greatest ease and in striking quantity the characteristic citryl-β-naphtocinchoninic-acid, melting at 384.8° to 386.6° F. As moreover the physical constants and the chemical reactions of the citral—splitting off of cymol, formation of the oxime, boiling point of the latter, etc. etc.—exhibited the most complete agreement, there could be no doubt of the identity of these compounds.

In reference to the preparation of large quantities of linalool, it has been observed that in linaloe oil, traces of methylheptenone (identical with that obtained by Wallach from cineolic acid) are always present, and that small quantities of geraniol in the free state frequently occur.*

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* Schimmel & Co's. Report, October, 1894.

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### *Anacardiaceæ.*

*Mastic oil.* By mastic is understood the fragrant resinous exudation from *Pistacia Lentiscus*, L., a small tree or shrub which is indigenous to the basin of the Mediterranean. It is chiefly obtained in a few islands of the Greek Archipelago, principally in Scio. The best quality of mastic is in globular, or more or less elongated, tears of the size of a pea, of a pale-yellow to dark-yellow color, transparent, of a glass-like lustre, and breaking readily with a conchoidal fracture. By distilling mastic with water about 2 per cent. of a yellowish volatile oil of a strong, agreeable odor is obtained. The oil is slightly dextro-rotatory, commences to boil at 311° F., and passes over almost entirely at 320° F. It is essentially a terpene, $C_{10}H_{16}$, which yields only with difficulty a crystallizing hydrochloride. The specific gravity of mastic oil is 0.858 at 59° F.; optical rotation +24°; yield of oil from the raw material 0.9 to 2.5 per cent.

*Cognac oil.* Although it is not definitely known whether this oil occurs ready formed in the grape (of *Vitis vinifera*, L., family *Vitaceæ*), or whether it is formed by fermentation, and though it is obtained in a peculiar manner, namely, from wine-lees, it may, on account of its importance, be here discussed.

The first statement regarding cognac oil was made by A. Rautert.* According to him the wine-lees (chiefly yeast separating in the fermentation of the must) were used for obtaining a kind of brandy or whisky. It was, however, noticed that the aroma of this brandy was due to a volatile oil, which, when added to spirits, imparted to them a cognac taste; on account of this property the volatile oil is now produced from the lees. For this purpose a barrel lined with lead is filled three-quarters full with wine-lees, three to six hundred-weights being taken for each operation. Through a rose upon the bottom of the barrel steam is introduced, the barrel being covered with a lid provided with an outlet-pipe for the vapors. This pipe is connected with a refrigerator. Distillation lasts 5 hours. The black drops of cognac oil covered by aqueous spirits of wine collect in the receiver. The residue resulting from distillation are used for obtaining argol. The oil as well as the aqueous spirits of wine has to be rectified, a further small quantity of oil being thereby obtained from the latter. The preparation of the lees before being brought into the barrel is of special importance. To every hundredweight of lees is added one to one and a half hundredweight of water and 8 ozs. of sulphuric acid, whereby the lees become less pasty and allow of an easier passage of the steam. Twenty-five hundredweight of lees yield about 1 lb. of cognac oil, or about 0.04 per cent. Pure cognac oil being very expensive, it is frequently adulterated with absolute alcohol.

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* Dingl. polyt. Jour., 143, 1857, S. 71.

By direct distillation of wine, frequently repeated, small quantities of cognac oil may also be obtained, 4000 parts of wine yielding about 1 part of cognac oil.

Cognac oil when rectified and clarified forms a colorless liquid of a strong, not agreeable odor. It is insoluble in water, but readily soluble in spirits, and imparts to the latter the odor and taste of cognac. Crude cognac oil sometimes possesses an acid reaction and has to be washed with soda solution; on heating, the resulting milky mixture again separates the oil, which is dried over calcium chloride.

According to Liebig and Pelouze, cognac oil is a mixture of free oenanthylic acid, $C_{17}H_{14}O_2$, and its ethyl ether. According to Dellfs, Liebig and Pelouze's oenanthylic acid is pelargonic acid, $C_9H_{18}O_2$. Later on Faget and Fischer proved that Liebig and Pelouze's oenanthylic acid is actually a mixture of capric and caprylic acids. Capric acid preponderates, however, to such an extent that cognac oil, may be considered to consist in the main of ethyl caprylate, $C_9H_{19}COOC_2H_5 = C_{12}H_{24}O_2$. This ester has a specific gravity of 0.862, and boils at 469.4° to 473° F.

*Artificial cognac oil* is obtained by the etherification of the volatile acids contained in cocoanut oil. The latter is a mixture of the most varying glycerides, among which capronic, capric and caprylic acids appear in not inconsiderable quantities. For the purpose of obtaining these acids the melted fat is saponified with soda lye, the resulting soap decomposed with sulphuric acid, and distilled with direct steam, whereby capronic and caprylic acids chiefly pass over and float in the form of an oily layer upon the water. This layer is separated from the water and converted with alcohol and sulphuric acid by gentle heating into ether. The latter is mixed with water, whereby it separates as an oily liquid, washed with carbonate of soda solution, and finally rectified in a current of steam. The cognac oil thus obtained closely resembles the genuine product, it only differing from it in that in a non-diluted state it has a less penetrating, disagreeable odor, but rather one of pine-apple.

Artificial cognac oil may also be obtained from rue oil, as well as from pelargonium oils. It is used not only for the production of cognac, but recently also for perfuming soap (in preparing the fruit aroma). The *cognac essence* of commerce is as a rule only a mixture of wine fusel oil with ethyl acetate and ethyl nitrate.

### *Euphorbiaceæ.*

*Cascarilla oil* (*Kaskarillöl,* G). Cascarilla bark is derived from *Croton Eluteria,* Benn., a low and even tree-like shrub indigenous to the Bahama Islands, and is used in medicine as well as in perfumery. It has a nutmeg-like odor and an aromatic, bitter, somewhat pungent taste. Its constituents are volatile oil, resin and a bitter principle—cascarillin. It is brought into commerce chiefly from Nassau, New Providence. For obtaining the oil the bark is comminuted, moistened with water, allowed to repose for 12 to 16 hours, and then distilled with steam. The yield is about 1 per cent. The oil is greenish or dark yellow, possesses a very pure aromatic odor and taste, and is quite thickly-fluid. Its specific gravity is 0.890 to 0.930 at 59° F. In alcohol it is quite readily soluble, and it commences to boil at 356° F. According to Völkel, cascarilla oil consists of two different constituents, one of which boils at 343.4° F., and is probably a terpene, whilst the other boils at a higher temperature, is quite thickly-fluid, possesses a less agreeable odor and very likely belongs to the oxygenated bodies. On the other hand, according to Gladstone, the oil is composed of two hydrocarbons, one of which smells like lemons and the other like calamus oil. Cascarilla oil dissolves in iodine without perceptible heating, and thickens thereby. Nitric acid colors the oil reddish, later on blackish-brown, and oxidizes it vigorously. Sulphuric acid gradually colors the oil red. In alcoholic lye the oil is readily soluble.

## VII. *Volatile Oils of the Umbelliferæ.*

The family *Umbelliferæ* comprises a large number of plants, the fruits, roots and frequently all parts of which contain volatile oil. In their physical properties the oils vary very much, even those from different parts of the same plant showing variations (water hemlock, heracleum, anise, angelica, etc.). Most of the oils are specifically lighter than water, but they become frequently heavier by the absorption of oxygen on exposure to the air. Many of them are optically active, and contain oxygen. According to their chemical constituents they may be grouped as follows: *Cuminol,* in the oils of water-hemlock and cumin; *anethol,* in the oils of anise and fennel; *carvol,* in the oils of caraway and dill (in the latter as a by-constituent); *coriandrol,* in coriander oil; further ester as a chief constituent in the oils of parsnip and heracleum; finally *terpenes,* as principal constituent in the oils of galbanum and carrot (dextropinene); in the oils of dill and sometimes of caraway (limonene); phellandrium oil (phellandrene); in the oils of parsley, mountain parsley, angelica and masterwort. Asafœtida oil stands by itself, it containing allyl sulphides as principal constituent, and hence belongs to the sulphuretted oils.

Of the greatest importance in a technical respect are the oils of anise, fennel and caraway, and next, the oils of dill, cumin and coriander.

*Water-hemlock oil* (*Wasserschierlingöl*, G). The seed of *Cicuta virosa*, L., the water-hemlock, when collected in the fall and distilled with water, yields a limpid, almost colorless, volatile oil, which is lighter than water, and possesses the odor and taste of cumin. The yield is 1.67 per cent. The oil from the seed contains cumin aldehyde or cuminol, $C_6H_4C_3H_7COH$, an oily liquid boiling at 457.9° F., and possessing the odor of cumin oil; further a hydrocarbon, probably cymol, $C_{10}H_{14}$, boiling at 348.8° F., and finally a very small brown residue. Hence the oil in its composition closely resembles cumin oil.

The root of water-hemlock, which is poisonous, also yields a volatile oil. According to Albrecht and Scheife, the oil is poisonous, which is, however, denied by Simon, though he admits the alcoholic extract of the root to be poisonous. A. H. van Ankum obtained from the root by distillation with water, 0.12 per cent. of oil, which showed a neutral reaction, dissolved only partially in 5 parts of alcohol of 0.84 specific gravity, but was miscible in all proportions with absolute alcohol, ether, chloroform, benzol, and carbon disulphide, and showed no poisonous properties. The principal mass of the oil consists of cicutene, a terpene $C_{10}H_{16}$, boiling at 330.8° F., having a specific gravity of 0.87038 at 64.4° F., and being dextro-rotatory. With hydrogen chloride a liquid combination is formed, while chlorine produces a viscous combination of the formula $C_{10}H_{12}Cl_4$, possessing a camphor-like odor.

*Parsnip oil* (*Pastinaköl*, G.). The fruit of *Pastinaca sativa*, L., the parsnip, contains a volatile oil which has been investigated by J. J. van Renesse. The oil obtained by distilling the ripe fruit with water is colorless, transparent, of a not disagreeable odor, and aromatic tase, and has a neutral reaction. Specific gravity 0.870 to 0.890 at 59° F. The yield, according to Schimmel & Co., is 2.4 per cent., and the known constituents of the oil, propionic-acid octyl ester and butyric-acid octyl ester.

*Heracleum oil* (*Herakleumöl*, G.). The fruits of the Heracleum species are rich in volatile oils. Thus far only the oils of *Heracleum sphondylium*, L., the cow parsnip of Europe, and *H. giganteum*, L., have been investigated.

*Oil of Heracleum sphondylium*, L. This oil was first investigated by T. Zincke. By distillation with steam of ordinary pressure, the fresh fruit yielded 0.3 per cent. oil. Like the fresh fruit, the water of distillation showed a highly acid reaction; it contained acetic and caproic acids, of the former more than 0.075 per cent. of the weight of the fruit. The oil was pale green, limpid, of a slight not disagreeable odor, and a pungent, acrid taste. Specific gravity 0.864 at 75.2° F. The oil showed a slightly acid reaction. It commenced to boil at 374° F., and passed over completely at 518° F. By fractional distillation were obtained: a portion of 11.67 per cent. of the oil boiling at 374° to 383° F., another portion boiling between 402.8° and 406.4° F.; and finally, at above 419° F., a third fraction, the principal mass of which boiled between 514.4° and 519.8° F. A by-fraction passed over between 419° and 514.4° F. In addition to other constituents not accurately determined, the fraction between 374° and 383° F., contained octyl alcohol $C_8H_{17}OH$, and possessed the peculiar, penetrating, aromatic odor of it. The second fraction (402.8° to 406.4° F.) had an agreeable orange-like odor and a pungent spicy taste; it had a specific gravity of 0.8717 at 60.8° F., and consisted of acetic acid octyl ester, $CH_3COOC_8H_{17} = C_{10}H_{20}O_2$. The octyl alcohol obtained therefrom had a specific gravity of 0.83 at 60.8° F., and boiling point 374° to 377.6° F.; it was a primary alcohol. The by-fraction, 419° to 514.4° F., and the principal fraction, 514.4° to 519.8° F., consisted of caproic acid octyl ester $C_5H_{11}COOC_8H_{17} = C_{14}H_{28}O_2$. The latter fraction was oily and without taste and odor. Schorlemmer proved the octyl alcohol of Heracleum oil to be primary normal alcohol.

W. Möslinger repeated the examination of the oil and arrived at the conclusion that Zincke's statements were not correct in all particulars, which probably was due to Zincke's oil having been prepared from over-ripe fruit. The oil prepared by Möslinger passed over completely between 230° and 555.8° F., somewhat more than 60 per cent. passing over between 397.4° and 402.8° F. and another 9 per cent. between 402.8° and 406.4° F.

Above 212° F. the pale-yellow color of the oil turned into red-yellow, and gradually became brown-red, the distillates remaining colorless. However, above 536° F. coloring matter also passed over and the residue underwent partial decomposition. On cooling the residue solidified; above 608° F. it consisted of lauric acid. The separate fractions were composed as follows:

230° to 347° F.: 0.56 per cent. butyric acid-ethyl ester, $C_3H_7COOC_2H_5 = C_6H_{12}O_2$.
347° to 397.4° F.: 4.5 per cent. mixture.
397.4° to 402.8° F.: more than 60 per cent., acetic acid octyl ester, $CH_3COOC_8H_{17} = C_{10}H_{20}O_2$.
402.8° to 410° F.: mixture.
410° to 464° F.: 1 per cent. of acetic acid octyl ester and caproic acid octyl ester.
464° to 527° F.: 3 per cent. caproic acid octyl ester, $C_5H_{11}COOC_8H_{17} = C_{14}H_{28}O_2$.
527° to 608° F.: same as above.

In 1875 fruit not entirely ripe was used for the preparation of the oil; it yielded only 0.84 per cent. The aqueous distillate contained as principal constituents acetic and caproic acids, and in addition methyl alcohol and very little ethyl alcohol. In the fraction from 176° to 338° F. only very small quantities of acetic ethyl ester and butyric acid ethyl ester were established, as well as small quantities of hexyl and octyl combinations (acetic acid ester?). Finally in the higher boiling portions capric acid octyl ester and lauric acid octyl ester in very small quantities were present.

From the ripe fruit Schimmel & Co. obtained a pale yellow oil having an acid reaction and a specific gravity of 0.80 to 0.87 at 59° F. It boils at between 176° and 572° F., and contains butyric acid ethyl ester, acetic acid ethyl ester, hexyl alcohol, ethyl alcohol, caproic acid octyl ester, and probably capric acid in the portion passing over between 527° and 608° F.

*Oil from Heracleum giganteum*, L. G. Gutzeit has prepared this oil by subjecting not entirely ripe fruit to distillation with three times its weight of water. The water of distillation contained 0.1 per cent. of the weight of the fruit of methyl and ethyl alcohols. The yield of volatile oil was 0.56 per cent. The three principal fractions were: 266° to 338° F. (10 per cent. of the total oil); 392° to 410° F.; and 410° to 482° F. The first fraction contained butyric acid ethyl ester. From ripe fruit Gutzeit obtained 2 per cent. oil, hence a considerably greater yield. It was, however, proved that the percentage of combinations with a low content of carbon was smaller in oil from ripe than from unripe fruit; with ripe fruit the fraction from 266° to 338° F. amounting only to 7.1 per cent. On the contrary the water in the distillation of unripe fruit contained preponderatingly ethyl alcohol, but in the distillation of ripe fruit chiefly methyl alcohol.

Heracleum oil is of special interest because it contains as chief constituents esters of the series of fatty bodies, viz., the oil of *Heracleum sphondylium* preponderatingly acetic acid octyl ester and next caproic acid octyl ester and the oil of *H. giganteum* acetic acid octyl ester and butyric acid hexyl ester. It also contains some other esters and apparently a few alcohols, which is also a rare occurrence.

*Aniseed oil, oleum anisi* (*Anisöl*, G.; *essence d'anis*, F.) The anise plant, *Pimpinella anisum*, L., contains volatile oil in all parts, but chiefly in the seed. The plant is an annual which is indigenous to the eastern portion of the Mediterranean basin and is extensively cultivated in many parts of Europe as well as in India and Chili, and occasionally in this country. The product from Southern Russia is considered the best. For the purpose of obtaining the oil the seed is thoroughly moistened with water and allowed to rest for 24 hours, it being several times turned over with a shovel during this time. It should not be comminuted, otherwise the oil escaping thereby from the cells will resinify. Distillation is best effected with steam; the condensers should not be cooled too much, since at 50° F. aniseed oil separates stearoptene which would cause the condensing pipes to be choked. From dry seed the yield amounts to from 2.4 to 3.5 per cent. In cleaning aniseed a waste is obtained, which is called *anise-chaff*, and yields only about 0.34 to 1.0 per cent. oil; it is richer in stearoptene than the actual aniseed oil. Finally it may be remarked that Schimmel & Co.

prepare by the cold way pure anethol, which is used as the strongest and best quality of aniseed oil. The residue resulting from the distillation of the seed is dried and forms an important fodder.

Aniseed oil is colorless to pale yellow; it is liquid at a higher temperature, but on exposure to cold becomes thickly-fluid to solid. A good quality of oil should solidify at from 41° to 59° F., and melt again at 42.8° to 64.4° F. However, if the oil be several times heated to its boiling point, it loses the capacity of crystallizing; also when very old, in which case it has also acquired a darker yellow color. The specific gravity of fresh oil is 0.98 to 0.995; that of old oil 1.0285. The specific gravity of a good quality of Mitcham and Hitchin oil ranges, according to Williams, from 0.976 to 0.984 at 60° F. The boiling point lies at 431.6° F.; according to Williams at 433.4° to 442.4° F. Aniseed oil is soluble in 5 parts of 90 per cent. alcohol and in 3.5 times the volume of petroleum-ether, a larger amount of the latter solvent giving a turbid mixture. It shows a neutral reaction, but resinifies readily in the air, and therefore should be kept in carefully closed bottles filled up to the neck, and in a dark place. The odor of the oil is anise-like and its taste agreeable, sweetish, and afterwards pungent. The oil is optically inactive or very slightly levo-rotatory.

The principal constituent of aniseed oil is formed by anethol (80 to 95 per cent.); it contains in addition an unknown liquid body. Anise-chaff oil is richer in solid constituents than aniseed oil; it resembling it otherwise, except that its odor is not quite so fine.

Iodine is taken up by aniseed oil without especially vigorous reaction, the absorption amounting, according to Williams, from 186 to 274 per cent., whereby the oil is gradually converted into a resin-like, brittle mass. Alcoholic hydrochloric acid colors the oil flesh color, the color, however, being at times only transitory. Nitric acid causes violent foaming, a mixture remaining behind from which water separates an orange-colored resin smelling of aniseed oil. An excess of sulphuric acid dissolves the oil with a red color. Chromic acid oxidizes the oil to anisic and acetic acids. Sodium dissolves clear in an ethereal solution of aniseed oil, a yellowish-white precipitate being formed only after standing for some time. According to Williams, the oil absorbs 0.25 to 0.38 per cent. caustic potash.

Aniseed oil is frequently adulterated with star anise oil. According to Umney, such sophistication is recognized partially by determining the "normal" solidifying point, and partially by the color produced by alcoholic hydrochloric acid, the former being from 49° to 55.4° F., with star anise oil, and the latter yellow-brown to brown. The effect of sodium (0.15 gramme) upon an ethereal solution of the oil (10 drops oil to 70 to 100 drops ether) may also be employed as a test, star anise oil yielding with this reaction a yellow sediment and a yellow solution.

Aniseed oil is also adulterated to a considerable extent with anise-chaff oil and the stearoptene of fennel oil. The latter sophistication is detected by the peculiar odor on heating the suspected oil.

According to Schimmel & Co.,* "normal" aniseed oil should at the time of purchase possess a melting point of not lower than 59° F. The method of testing to be adopted is as follows:

"Reduce the temperature of the sample of oil under examination by immersing it in a freezing-mixture; then add, with a rod, a trace of crystallized oil. The entire mass should thereupon solidify to the consistency of a crystalline paste, which should not again become liquid below 59° F. Care is to be taken that the oil in the sample selected for examination be completely liquid and uniformly mixed."

Aniseed oil is used in perfumery, in the preparation of liqueurs and for medicinal purposes. Small quantities of it mixed with other volatile oils give good perfumes for soaps and pomade, but it is not suitable for handkerchief perfumes.

In conclusion it may be remarked that anethol is brought into commerce as a substitute for aniseed oil. It has a specific gravity of. 0.986 at 77° F. and melts at 69.8° to 71.6° F. In contact with air a small portion of it becomes oxidized, whereby the specific gravity is increased and the melting point lowered.

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* Report, October, 1894,

*Pimpinella oil, oleum pimpinellæ* (*Pimpinellenöl*, G.). The roots of the small burnet saxifrage, *Pimpinella Saxifraga*, L., and *P. magna*, L., yield by distillation with water a golden-yellow, limpid, very volatile and specifically light oil, of a disagreeable penetrating parsley-like odor, and a bitter, acrid taste. By nitric acid it is converted into a brown resin. It has a specific gravity of 0.959 at 59° F., and commences to boil at 464° F. The boiling point rises to 572° F., when products of decomposition pass over. The yield is 0.025 per cent.

The root of *Pimpinella nigra*, Willd., yields by distillation with water a beautiful pale-blue oil; however, on standing, the color passes into green. The oil has a burning sharp and acrid taste and a slighter odor than the above-mentioned pimpinella oil. By nitric acid it is also converted into a brown, odorless resin.

*Fennel oil, oleum foeniculi seminis* (*Fenchelöl*, G.; *essence de fenouil*, F.) is obtained by distillation with steam from the fruit of the garden fennel, *Foeniculum officinale*, All. = *F. capillaceum*, L., as well as from the fruit of the sweet fennel, *Foeniculum dulce*, D. C., the latter having larger seeds that the former and is cultivated only in southern countries. Abyssinia and Persia appear to be the home of the fennel, but it is frequently found wild in Western and Southeastern Europe and in Western Asia. It is especially cultivated in France, India and China, and more recently in Germany, Galicia, Roumania and Japan. For distilling it is best to use fresh green fruit which, without being comminuted, is moistened with water, allowed to repose for 12 hours, and then distilled with steam. The temperature of the cooling water should not be below 59° F., otherwise stearoptene separates from the oil. The yield is 3 to 7 per cent., being, as a rule, about 5 per cent. The dried residue from distillation forms an excellent fodder.

Crude fennel oil is frequently dark yellow, but when rectified is colorless or slightly yellowish; by age it gradually becomes darker. It has a fennel-like odor and a sweetish, mild, aromatic taste. The specific gravity ranges from 0.94 to 1.0, but is generally 0.96 to 0.99; it appears to increase with age. The oil solidifies at 50° F., though there are fennel oils which remain liquid at 0.4° F; the rectified oil solidifies with greater difficulty than the crude product. The principal mass of the oil distils off between 248° and 437° F. In 1 to 2 parts of 90 per cent. alcohol fennel oil dissolves to a clear liquid. An oil of less value is also obtained from the herb of the fennel plant.

The various fennel oils consist of pinene, phellandrene, dipentene, fenchone, anethol. But in none of them do all constitcents appear simultaneously. Certain oils, for instance, do not contain phellandrene, others no fenchone, others again no anethol. The optical rotation varies from +7° to +22°.

In its chemical reactions fennel oil resembles aniseed oil, it being only necessary to take into consideration its smaller content of solid anethol and its content of phellandrene.

Fennel oil is adulterated with oil of turpentine and alcohol. Besides an oil is frequently brought into commerce from which the larger part of anethol has been extracted. This oil possesses a bitter taste and is, of course, of less value.

In Moravia, according to E. Campe, Galician or Polish fennel oil is subjected to fractional distillation, oil as old as possible being used. At first an oil of a slightly sweet taste passes over, and then a limpid oil of a bitterish, disagreeable acrid taste. Then follows a sweet distillate smelling of fennel, which is sold as I *a* fennel oil. Finally anethol passes over, whereby the fraction acquires an anise-like taste. As a rule the oil yields 12 to 16 per cent. elaeoptene (the first distillates), 12 to 14 per cent. so-called I *a* fennel oil and 60 to 70 per cent. stearoptene (anethol). The first running is exposed to the air for 3 or 4 months, and then once more subjected to fractional distillation, whereby more anethol is obtained. The stearoptene is also for some time exposed to the air so that it entirely loses its fennel odor. Mixed with fennel oil it is used for perfuming soap and in the sophistication of rosemary oil.

Fennel oil is chiefly used in the manufacture of liqueurs, but also for scenting soaps and aromatic waters.

*Phellandrium oil, oleum phellandrii* (*Wasserfencheöll,* G.) The fruit of the five-leaved water-hemlock, *Phellandrium aquaticum,* L., is moistened with water, and distilled with water or steam 12 to 18 hours, whereby it yields 0.8 to 1.5 per cent of volatile oil. Phellandrium oil is yellow, limpid, possesses the peculiar odor of the plant (different from fennel), and an aromatic, slightly bitterish taste. Its specific gravity is 0.80 to 0.89 at 59° F. It dissolves in 3 to 4 parts of alcohol, as well as in equal volumes of ether.

L. Pesci has subjected the oil to a thorough investigation. By fractional distillation he obtained from it a terpene, $C_{10}H_{16}$, boiling at 339.8° to 341.6° F., which he called *phellandrene.* Its specific gravity is 0.8558 at 50° F. By heating in a closed tube to between 284° and 302° F. a solid, non-crystallizing body, probably a diterpene, $C_{20}H_{32}$, is formed. Phellandrene forms a nitril $C_{10}H_{16}N_2O_3$ melting at 197.6° F.

The results obtained by Pesci were later on confirmed by Wallach's investigation, so that the hydrocarbon of both fennel oil and phellandrium oil is phellandrene, the difference between the two oils being that phellandrene forms the chief constituent of phellandrium oil. The latter when taken internally is said to be poisonous, but phellandrene is not, so that the poisonous properties of the oil must be due to an unknown constituent.

*Dill oil, oleum anethi* (*Dillöl,* G., *essence d'aneth,* F.), is obtained by distillation with water from the fruit of the dill, *Anethum graveolens,* L. The fruit, and frequently the entire herb, is moistened with water, allowed to lie for some time, and then distilled. From the fruit the yield is 2.5 to 4 per cent. Dill is indigenous to Southern Europe, Asia Minor, and is cultivated in other parts of Europe, as well as in this country, and in India.

Dill oil is pale yellow, after long keeping reddish brown. It has a peculiar penetrating odor of dill, and at first a warming, sweetish taste, which later on becomes pungent and acrid. Its specific gravity ranges from 0.905 to 0.915. Optical rotation +70° to 80°. It dissolves in 10 parts of absolute alcohol and the same quantity of ether. It is also soluble in 1500 parts of water, such solution forming the commercial dill water.

R. Nietski* instituted a series of experiments in order to determine the chemical character of this oil. He found that traces of an acid having all the reactions of butyric acid pass over during the distillation of the oil. The oil, when subjected to fractional distillation, yielded three distinct substances, two of them hydrocarbon, the third an oxygenated oil. The latter was determined to be identical with *carvol*, and amounted to 30 per cent. The two hydrocarbons are terebenes of composition $C_{10}H_{16}(O = 16)$. One, amounting to 10 per cent., boils between 311° and 320° F.; the other, amounting to 60 per cent., at 338° to 347° F. Both of them were capable of being converted into cymol. In addition to carvol, O. Wallach established in dill oil the presence of limonene, boiling between 347° and 354.2° F., which yielded the characteristic tetrabromide, melting at 219.2° to 221° F.

Hence the principal constituent of dill oil is limonene (at least 60 per cent.), while in addition it certainly contains carvol (30 per cent.), and probably another terpene (10 per cent.) East Indian dill oil contains, according to Schimmel & Co.,† an ingredient which is heavier than water.

Iodine is dissolved in dill oil, with moderate reaction, to a red-brown liquid. Cold nitric acid colors the oil darker, and, on heating, converts it into a yellow resin. Nitric acid colors dill oil red brown.

Dill oil is used in medicine and in perfumery. Mixed with other volatile oils it forms a good perfume for soap.

*Caraway oil*, *oleum cari* or *carui* (*Kümmelöl*, G.; *essence de carvi*, F.). The common caraway seed of commerce consists of the fruit split into the two mericarps of the caraway plant, *Carum Carui* (*Carvi*), L. The caraway plant is indigenous to the Western Himalayas and the Caucasus, and is found throughout Siberia and the greater portion of Europe, but is scarce in the southern part of that continent. It is extensively cultivated in Norway, Russia, Germany, Holland, England, Morocco, and also in the United States. The yield of fruit per acre is between 8 and 10 cwt. The Holland product is the best, while Swedish, Norwegian and other varieties of seed are not suitable for obtaining volatile oil.

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* Archives of Pharmacy, April, 1874.

† Report, October, 1893.

Caraway oil is of considerable technical importance. It is obtained by distillation with steam, the seed being previously soaked. Schimmel & Co. of Leipsic, work annually from 88,000 to 110,000 lbs. of caraway into volatile oil. The retorts used hold 5500 lbs. of seed; distillation lasts 6 to 8 hours. The residue constitutes an excellent fodder. The yield may be increased by comminuting the seed, but by allowing the seed to lie in a comminuted state the quality of the oil is injured.

The yield of the oil varies very much according to the derivation of the seed. The same applies to the quality of the oil, which is dependent on the content of carvol. Generally speaking the limit of yield is from 2.9 to 7 per cent. Dutch seed yields 4 to 6 per cent. of a good quality of oil, and German cultviated seed 4 to 4.3 per cent. The wild seed brought into commerce from East Prussia and Finland, to be sure, yields 4 to 7 per cent. of oil, which, however, is of inferior quality. The same applies, according to C. Nicolaysen, to Norwegian seed, which yields 6.1 to 6.4 per cent. of oil, the latter, however, containing only 47 to 49 per cent. carvol. Generally speaking the yield of oil from plants cultivated in northern localities is better than from plants of southern derivation. The quantity of oil in the fruit also increases with colder, moist weather. The degree of ripeness appears to be of less influence upon the yield.

*Caraway-chaff oil* is obtained by distillation with steam from the herb of the plant, the chaff produced in threshing the fruit being also used. The oil is of inferior quality.

Caraway oil, when fresh, is of a yellow color, but soon turns dark yellow to brownish, especially when exposed to the light, the change taking place the more rapidly the less pure the oil is. The oil has a pure caraway odor and a pungent, aromatic taste. Its specific gravity ranges from 0.905 to 0.915 at 59° F. It commences to boil between 347° and 374° F.; at 473° F. a brown, resinous mass remains behind. It dissolves in equal volumes of 90 per cent. alcohol and with the same ease in ether. Optical rotation, + 75° to + 85°.

Caraway-chaff oil has also a caraway-like odor, but less pure and agreeable; its taste is acrid and resinous.

Caraway oil consists of carvol, $C_{10}H_{14}O$ (60 to 70 per cent. in Dutch oil, 45 to 50 per cent. in German and Norwegian), and limonene, $C_{10}H_{16}$ (40 to 35 and 55 to 50 per cent). The characteristic odor of the oil is due to carvol, on the content of which its quality also is dependent.

In its chemical reactions caraway oil closely resembles dill oil, this being due to the analogous chemical composition of the two oils. Iodine, according to Williams, 258 to 263 per cent., is absorbed with considerable heating, the carvol being thereby converted into carvacrol; the same conversion of carvol is caused by phosphoric acid, caustic potash, etc. Bromine yields limonene tetrabromide; sulphuretted hydrogen the crystalline combination with carvol. Caustic potash is absorbed to the extent of 0.35 to 0.53 per cent., while on heating a considerable quantity of potassium is taken up, hydrogen being evolved. Sulphuric and nitric acids convert the oil into a smeary black resin.

Caraway oil is adulterated with oil of turpentine, alcohol, caraway chaff oil, etc., a mixture of oils of turpentine and caraway oil being even brought into commerce as caraway-chaff oil. A content of oil of turpentine, however, is readily detected by the odor. Frequently caraway oil from which the principal quantity of carvol has been withdrawn is sold at a low price; such oil has a specific gravity below 0.91. The carvol of commerce is frequently nothing but caraway oil freed as much as possible from carvene by fractional distillation. At 68° F. 1 part of pure carvol dissolves in 16 to 17 parts of 50 per cent. alcohol; in the presence of carvene (terpene) the solution appears turbid.

Caraway oil is used in pharmacy as an addition to purgative medicines to prevent griping, in the manufacture of liqueurs, in perfumery and for scenting soaps.

*Ajowan oil.* (*Ajowanöl*, G.) The fruit of *Carum Ajowan*, Benth. = *Ptychotis Ajowan*, D. C., contains a volatile oil, which is of interest on account of being extremely rich in thymol. The plant is indigenous to the East Indies. In preparing the oil large crystals separate from the aqueous portion of the distillate. These crystals are sold in Posnah and other large cities of the Deccan under the name of *Ajowan Ka Phul* (*Ajwain Kaphyl*) *i. e.*, sublimed ajowan. The plant is well known in the East Indies on account of its aromatic and medicinal properties. The seed especially contains volatile oil; it resembles caraway, but is smaller and smells of thyme oil. By repeated distillation with water the yield of oil amounts to 3 to 4 per cent. of the weight of seed. The oil is chiefly prepared in Oojein.

Ajowan oil is of a pale brown color and an agreeable aromatic odor. Its specific gravity is 0.900 to 0.930 at 59° F.; it commences to boil at 320° F., the boiling point gradually rising to 428° F. The distillate passing over at the latter temperature crystallizes on cooling, the residue (⅓ to ¼ of the weight of crude oil) being also converted, after standing, into a crystalline mass. By allowing ajowan oil to stand in an open dish, beautiful crystals of stearoptene separate out, corresponding to the gradual evaporation.

According to Stohmann, ajowan oil consists of 30 to 40 per cent. thymol, $C_{10}H_{14}O$, 30 to 40 per cent. thymene, $C_{10}H_{16}$, 15 to 20 per cent. cymol, $C_{10}H_{14}$, and small quantities of a liquid phenol (probably carvacrol). The thymene boils at 321.8° to 329° F., and has a specific gravity of 0.868. Generally speaking, ajowan oil resembles thyme oil, but is essentially richer in thymol. The latter may be obtained from the oil by shaking with soda lye, whereby the thymol dissolves in the lye. On neutralizing the solution with sulphuric acid, the thymol reseparates, and after distilling with steam, is repeatedly crystallized from alcohol. The only known use of ajowan oil is for the preparation of thymol.

*Cumin oil, oleum cumini* (*Römisch Kümmelöl,* G.; *essence de cumin,* F.). The cumin plant, *Cuminum cyminum,* L., is indigenous to Egypt and other parts of Africa, and is cultivated in various parts of Asia and Europe. The seed is brought into commerce chiefly from Spain, Sicily, Syria, Morocco and India, the best variety coming from Malta. For the purpose of obtaining the volatile oil, the seed is moistened, allowed to lie for some time, and then distilled with steam. The yield amounts to 2.8 to 3.8 per cent. The oil is chiefly contained in the pericarp of the fruit, to a less extent in the actual seed.

. Cumin oil is colorless to yellow, as a rule yellow, and the darker the older it is. It is limpid, but becomes more thickly-fluid by age and acquires thereby an acid reaction. Its odor is disagreeably penetrating like that of the plant, and its taste very sharp, aromatic, and somewhat bitterish. The specific gravity of the oil ranges from 0.890 to 0.930 at 59° F. The oil commences to boil at 338° F., the boiling point gradually rising to over 446° F. It is soluble in water to a comparatively large extent, and in all proportions in absolute alcohol. Of alcohol of 0.85 specific gravity it requires 3 parts at 59° F. It consists of about 33 per cent. cymol and 77 per cent. cuminol or cuminaldehyde.

The most important chemical reaction of the oil is that it gives with $NaHSO_3$ crystalline separations. Iodine dissolves readily and with moderate heating in the oil, a liquid yellow-brown mass being formed. The oil is vigorously attacked by chlorine and bromine, substitution-products of cuminol ($C_{10}H_{11}ClO$ and $C_{10}H_{11}BrO$) being formed. Nitric acid converts the cuminol into white crystals of cumic acid. Sulphuric acid colors the oil deep red; on adding water a slimy mass of a dirty color is separated.

Cumin oil is chiefly used for the preparation of cymol. cuminol being obtained as a by-product. Formerly the oil was also used in medicine, for instance, as a remedy for spasms in the stomach.

*Parsley oil* (*Petersilienöl*, G). The fruit of the parsley, *Petroselinum sativum*, L., = *Apium Petroselinum*, contains a volatile oil which is obtained by distilling the moistened fruit with water. Distillation has to be continued for quite a long time, because half the oil consists of stearoptene, which volatilizes with difficulty. The yield is about 2.4 per cent. German parsley yields the best oil; though East Indian oil is also found in commerce. Parsley oil is colorless to pale greenish-yellow, but becomes darker by age. It is thickly-fluid, possesses a strong odor of parsley, and a mild, aromatic, later on pungent taste. Its specific gravity is 1.07 at 59° F. The principal mass of the oil boils between 320° and 410° F., and another portion between 518° and 554° F. The oil has a neutral reaction and dissolves readily in alcohol and ether. By shaking with water it may be decomposed to a limpid, light oil and a thickly-fluid heavy oil, the latter crystallizing in the cold. The unchanged oil also separates stearoptene on exposure to cold, and even solidifies entirely. Parsnip oil consists of a terpene, $C_{10}H_{16}$ (boiling point, 320° to 327.2° F.; levo-rotatory), and a stearoptene—apiol, $C_{12}H_{14}O_4$. It is only rarely used as a diuretic. Schimmel & Co. furnish apiol in beautiful acicular crvstals, melting at 86° F.

*Athamanta oil* (*Bergpetersilienöl*, G.). The fresh herb of *Athamanta Oreoselinum*, Mönch., yields by distillation with water a volatile oil of a strong, aromatic, somewhat juniper-like odor, which boils at 325.4° F. and has a specific gravity of 0.843. By fractional distillation two principal portions are obtained. Both possess the composition of a terpene, $C_{10}H_{16}$; only a very small quantity of an oxygenated body could be established. With hydrogen chloride the terpene does not yield a crystallizable product, rather a liquid hydrochloride, $C_{10}H_{16},HCl$, boiling at 374° F.

*Celery oil, oleum apii* (*Sellerieöl*, G.). All parts of the celery plant, *Apium graveolens*, L., contain volatile oil, the best quality, however, being obtained from the seed. It is of a colorless to pale-yellow color, penetrating odor and sweetish taste. Its specific gravity is 0.889 to 0.890 at 59° F. The oil, according to Stohmann, is chiefly prepared for the Paris market. The yield from the seed is 2.5 to 3.5 per cent. Optical rotation, +67°4′. The oil is reputed to possess aphrodisiacal properties. In France the oil is distilled only from wild plants, about 400 lbs. of which yield 1 pound of oil.

*Angelica oil, oleum angelicae* (*Engelwurz- or Angelikaöl*, G.; *essence d'angélique*, F.). The fruit as well as the root of the angelica plant, *Archangelica officinalis*, Hoffm., = *Angelica Archangelica*, L., = *Angelica officinalis*, Guibourt, contains a volatile oil of a characteristic odor, which possesses not only a certain technical value, but is also of scientific interest. The plant grows in moist localities in Central Europe; the best roots being brought from the Saxonian Erzgebirge and from Thuringia. Recently Japanese angelica root is also brought into commerce. The oil usually used is obtained from the root, but that from the fruit (seed) is of finer quality. From the root the oil is best obtained by distillation with steam, the fresh roots having previously been cut in small pieces. From the seed the oil is obtained in a similar manner. The yield from the seed is 1.15 per cent. and from the root 0.75 to 1 per cent.

*Angelica root oil* is a mobile liquid, colorless when fresh, becoming yellow by exposure to the light. It has a strong, aromatic odor of the root and a pungent, aromatic taste. Its specific gravity ranges from 0.855 to 0.905 at 59° F.; the boiling point of the principal mass lies between 320° and 347° F. Optical rotation, +26° 45′ to +30° 7′. When exposed to the air the oil absorbs oxygen and gradually resinifies.

*Angelica seed oil* possesses an odor similar to that of root oil, but much finer. In a fresh state it is limpid and of an amber-yellow color. Its specific gravity is 0.8549 at 59° F. The oil is dextro-rotatory. Older oil is somewhat thickly-fluid, of a brown color and specifically heavier (specific gravity 0.9086 at 59° F.) Physically the principal difference between the two oils is the odor.

Angelica root oil contains only a terpene (75 per cent.; boiling point 348.8° F.) and a small quantity of bodies polymeric with it, the latter being formed during distillation in consequence of the heat. Old thickened oil will also contain such polymeric terpenes.

Angelica seed oil contains a terpene boiling at 342.4° to 347° F. perhaps a body isomeric with it, and further methylethyl acetic acid and oxymyristic acid.

Angelica oil is occasionally adulterated with oil of turpentine. It is chiefly used in the preparation of liqueurs.

*Lovage oil* (*Liebstocköl*, G.; *essence du livèche*). The root of lovage, *Levisticum officinale*, Koch. = *Ligusticum Levisticum*, L., when dried, comminuted and distilled with steam, yields a yellow brownish thickly-fluid volatile oil possessing the odor and taste of the root. The oil is readily soluble in alcohol, and by concentrated sulphuric acid is colored dark brown red, and by nitric acid, red. The yield is 0.6 to 1 per cent. from the dried root and 0.38 per cent. from the fresh root. Specific gravity of the oil 1.03 to 1.04 at 59° F. The oil is frequently adulterated with orange oil, and oils of copaiva and turpentine. The fruit also yields about 1.1 per cent. of a volatile oil having a specific gravity of 0.935 at 59° F.

*Sumbul oil* (*Sumbul- or Moschuswurzelöl*, G.) There are several varieties of sumbul root. The best kind is derived from the sumbul plant, *Euryangium Sumbul*, Kauff. = *Angelica moschata* Wigg. = *Ferula Sumbul*, Hook. fil., indigenous to Turkestan, Bucharia, Eastern Siberia, and probably to other parts of Central Asia. In India the root of *Dorema Ammoniacum*, Don., is used as sumbul root; it differs from the genuine root and is of less value. Sumbul root has a musk-like odor and an aromatic, very bitter taste. It is used in medicine as well as in perfumery. By extracting it, according to Reinsch, with ether, and the ethereal solution with alcohol, a clear pale yellow resinous residue (sumbul balsam) remains behind on evaporation. This resin contains an abundance of angelic and valerianic acids. By subjecting it to destructive distillation, a blue oil passes over, which contains umbelliferon, $C_9H_6O_3$ (Husemann). Schimmel & Co. separate by distillation from the root a volatile oil of specific gravity 0.954 at 59° F. The yield is 0.18 to 0.38 per cent. The price of the oil depends on the supply of root from Russia by way of which it enters commerce. Thus in 1889, when the supply was limited, the price of 1 kilogramme (2.2 lbs.) of sumbul oil was 600 marks ($150.00). According to Piesse sumbul root is especially used in Russia.

*Ammoniacum oil* (*Ammoniakgummiöl*, G). Ammoniac is a gum-resinous exudation from *Doremus Ammoniacum*, Don. When subjected to distillation with water it yields 0.3 to 0.4 per cent. of a colorless oil, possessing a powerful odor. The oil is slightly dextro-rotatory; specific gravity 0.891 at 59° F.; boils between 482 and 554° F. Nothing further is known of the composition of the oil than that it is free from sulphur. The root yields fictitious sumbul oil.

*Asafœtida oil* (*Stinkasant- or Asafœtidaöl*, G). Asafœtida is a gum-resin obtained by incision, from the living root of *Ferula Scorodosma*, B. et A., and *Ferula Narthex*, Boiss., both large perennial herbs, the former indigenous to Turkestan, Bokhara, and Western Afghanistan, and the latter to Western Thibet and probably to Cashmir. For the purpose of obtaining volatile oil, the gum-resin is comminuted, distilled with water from glass retorts in a common salt bath, and the escaping vapors are condensed in glass coolers. The yield is from 3.3 to 3.7 per cent. The oil is clear, limpid, pale yellow, has a penetrating odor of asafœtida and at first a mild, later on an acrid, taste. It has a neutral reaction and does not redden the skin. It is quite soluble in water and readily in alcohol and ether. On standing, sulphuretted hydrogen is constantly evolved. On exposure to cold the oil does not separate stearoptene. It boils at 275° to 284° F., but is decomposed by boiling. When exposed to the air it absorbs oxygen, becomes acid, and acquires a different odor. Specific gravity of the oil, 0.985 at 59° F.; optical rotation —9° 15′.

Asafœtida oil consists of allyl sulphide, allyl disulphide, terpenes, sesquiterpenes and blue oil.

*Galbanum oil* (*Mutterharz- or Galbanumöl*, G.). Galbanum is a gum-resin derived from *Ferula galbaniflua*, Boiss. and *F. rubicaulis*, Boiss. the former indigenous to Northern Persia, and the latter to Southern Persia. It has a penetrating odor and a bitterish, pungent taste. When subjected to distillation with 4 parts of water a colorless to yellowish oil passes over which has a specific gravity of 0.910 to 0.930 at 59° F., boils at 320° to 338° F., is dextro-rotatory, has an odor of galbanum and camphor, and a bitter, aromatic taste, which at first is pungent and then cooling. The yield of oil is 14 to 22 per cent.

O. Wallach has established in galbanum oil the presence of sesquiterpene, $C_{15}H_{24}$, it being found in the portions boiling between 518° and 536° F. By saturating the ethereal solution of the sesquiterpene with hydrogen chloride it yields the characteristic dihydrochloride, $C_{15}H_{24},2HCl$, which melts at 320° to 329° F. Galbanum oil may be said to consist of pinene (dextro-pinene) boiling between 320° and 325° F., and sesquiterpene boiling between 518° and 536° F., the former constituting the chief portion. By nitric acid galbanum oil is converted into a yellow resin, and by sulphuric acid it is colored violet, vigorous reaction taking place thereby. Galbanum oil is used internally, as well as externally, for embrocations and salves.

*Opoponax oil* (*Opoponaxöl*, G.). The opoponax plant, *Ferula Opoponax*, L., = *Opoponax Chironium*, Koch., is a perennial herb indigenous to Southern Europe. On wounding the root or lower part of the stem, a yellowish milky juice exudes which after hardening constitutes opoponax. This gum-resin is reddish-yellow, has a strong odor and a bitter, pungent taste. According to Pelletier, it contains a volatile oil of a peculiar, powerful odor. According to Przeciszewski, the oil is greenish-yellow, becomes darker in the air and is free from sulphur. According to Schimmel & Co., the gum-resin yields 6.5 to 8.5 per cent. of oil having a specific gravity of 0.860 to 0.891 at 59° F. and boiling between 392° and 572° F. The portions boiling at the lower temperature contain the odoriferous constituent. Opoponax oil is used in perfumery.

*Carrot oil* (*Möhrenöl*, G.). The fresh root of the carrot, *Daucus Carota*, L., contains a small quantity of oil which has been obtained by Schimmel & Co. by distillation with steam. The oil is yellow, of a pungent odor, and agreeable carrot-like taste. It dissolves in alcohol, glacial acetic acid, ether and chloroform, and has a specific gravity of 0.8829 at 68° F. On account of its content of acetic acid it shows an acid reaction; it does not solidify at 5° F. It commences to boil at 212° F., and yields two principal fractions, one between 275° and 329° F., the other between 413.6° and 428° F. From these fractions two portions may be isolated by rectification, one between 311° and 329° F., the other between 413.6° and 446° F. The former is water-clear, smells of the crude oil, has a neutral reaction, and a specific gravity of 0.8525 at 68° F.; it consists of dextropinene, $C_{10}H_{16}$, or a body closely allied to it. The second fraction amounts to about 40 per cent. of the crude oil; it is pale yellow, of a slightly aromatic taste, and has a specific gravity of 0.9028 at 68° F. This fraction is closely allied to cineol, $C_{10}H_{18}O$, and possesses its composition.

*Masterwort oil* (*Meisterwurz- or Imperatoriaöl*, G.). The masterwort, *Imperitoria Ostruthium*, L., iudigenous to the mountainous regions of Southern and Central Europe, has a root-stock of aromatic odor which is brought into commerce under the name of masterwort. It contains about ¾ per cent. of volatile oil which, according to Hirzel, is a mixture of several hydrates of a hydrocarbon, $C_{10}H_{16}$, and is obtained by distilling the bruised root with water. The oil being quite soluble in water, it is best shaken with ether, the ethereal layer separated, and the ether distilled off. The crude oil is dark brown and has to be carefully rectified with water, whereby a tar-like residue remains behind. The pure oil is colorless, water-clear, limpid, of an aromatic odor, and a pungent, warming taste. Its specific gravity is 0.877 at 59° F.; it boils between 338° and 374° F. By heating the oil with platinum chloride an odor of angelic acid becomes apparent, and Wagner therefore supposes the presence of angelic acid aldehyde $C_5H_8O = C_4H_7COH$ in the oil.

*Coriander oil, oleum coriandri* (*Korianderöl*, G.; *essence de coriandre*, F.) is obtained from the fruit of the coriander plant, *Coriandrum sativum*, L., probably indigenous to China and to the eastern portion of the northern shore of the Mediterranean, but is now extensively naturalized in the fields of temperate Asia and Europe. It is occasionally cultivated in the United States, and on a large scale in France, England and Germany, as well as in Morocco, but chiefly in India. In an unripe state the fruit, like the entire plant, possesses a bug-like, somewhat narcotic odor, while in the ripe state it has an aromatic taste and odor. For the purpose of obtaining the oil, the fruit is moistened with water, allowed to rest for 12 to 16 hours, and then distilled; or the fruit is bruised or ground and brought together with water into the retort. The yield amounts to 0.2 to 1 per cent. according to the derivation of the seed.

Coriander oil is almost colorless, has the odor and taste of the fruit, and a specific gravity of 0.874 to 0.882 at 15° F. Optical rotation + 4° to + 13°. It commences to boil at 300° F., the principal mass distilling over up to 392° F. It dissolves readily in alcohol, glacial acetic acid and ether.

The chief constituent of coriander oil is an isomeric borneol —coriandrol, $C_{10}H_{18}O$— it containing in addition some dextropinene, the quantity of which is estimated at about 5 per cent.

According to Barbier,* coriandrol constitutes a dextrorotatory variety of linalool. With the exception of their rotatory power, in which they are opposed to one another, both bodies possess the same physical properties, and also show themselves identical in their chemical attributes. Upon oxidation they yield the same aldehyde (citral), separate water in the same manner, and, finally, may both be converted into geraniol by a definite process.

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* Comptes rendus, CXVI (1893), p. 1459.

With iodine coriander oil decomposes with explosive phenomena. Iodine tincture colors the oil green, later dark green; very dilute nitric acid colors it raspberry red and, on heating, lilac and blue, both colorations disappearing on the addition of orange oil or alcohol. Sulphuric acid colors the oil brown-red; fuchsin is not dissolved by it.

Coriander oil is frequently adulterated, the adulterant most frequently employed being sweet-orange oil. The property of coriander oil to dissolve in dilute alcohol offers a good starting point to judge of its purity. Genuine oil should yield a clear solution when mixed with 3 parts of 70 per cent. alcohol at 68° F., additions of oil of turpentine, cedar-wood oil, etc., betraying themselves by their insolubility.*

### VIII. *Volatile Oils of the Families Saxifragaceæ, Myrtaceæ, Rosaceæ, Drupaceæ, Papilionaceæ, and Cæsalpiniaceæ.*

This division embraces a series of important volatile oils, which are obtained from the leaves of the plants, partially from the flowers and fruits, and finally also from the balsam exuding from the trunks. All the oils (perhaps with the exception of copaiva oil) contain oxygen. There is found in them especially *cineol* (oils of cajeput, checken leaf, myrtle and eucalyptus); *eugenol* (oils of pimento and cloves); *aldehydes* (oils of spiræa, bitter almonds, cherry laurel); further oils of cajeput, common eucalyptus (Peru balsam); *esters* (oils of storax, Peru balsam and tolu balsam). *Terpenes* occur in the oils of cheken leaf, myrrh and common eucalyptus (pinene); further in Australian eucalyptus oil (phellandrene), in oils of pimento and cloves (sesquiterpene), in oils of cajeput and tolu balsam, and finally in copaiva oil (diterpene).

It may be mentioned that the volatile oils of bitter almonds, cherry laurel and spiræa do not occur ready-formed in them, but are formed by the decomposition of glucosides. Physically, the oils to be discussed here show great variations. The oils of cajeput, myrtle, rose and copaiva are specifically lighter than water, the rest being heavier.

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* Schimmel & Co., Report, October 1893.

### *Saxifragaceæ.*

*Storax oil* (*Storaxöil*, G). Storax is a balsam prepared from the bark of *Liquidambar orientale*, Mill., a tree indigenous to the south-western part of Asia Minor, where it forms forests. The tree closely resembles the sweet-gum tree of North America, from which it differs in having smooth leaves with obtuse, often three-lobed and finely serrulate lobes, and in producing smaller heads of fruit. To obtain the balsam the outer bark, according to Campbell, is stripped off, the inner bark scraped off with a semicircular or sickle-shaped knife, and boiled in water upon which the resinous matter comes to the surface and is skimmed off. The boiled bark is next put into hair-sacks and pressed, boiling water being added to assist in the extraction of the resin. Liquid storax smells at the same time of jonquil and tar oil, but when diluted with other substances, only the first-mentioned agreeable odor remains behind. By extracting the storax with alcohol, storax-tincture used in perfumery is obtained.

By distilling liquid storax with water a volatile oil is obtained which consists of styrol and several cinnamic acid esters. Notwithstanding its comparatively high price it is frequently used in perfumery as a substitute for liquid storax. The yield is 0.3 to 0.7 per cent. Specific gravity of the oil, 0.89 to 0.90 at 59° F.

### *Myrtaceæ.*

*Cajeput oil, oleum cajeputi* (*Kajeputöl*, G., *essence de cajaput*, F.) The cajeput tree, *Melaleuca Leucadendron*, L., indigenous to the Moluccas and Sunda islands, contains in its leaves, bark and branches a volatile oil which is obtained by distilling the comminuted branches together with the leaves and water. The oil is also obtained from other varieties of *Melaleuca*

(*M. minor*, Smith; *M. Cajeputi*, Roxb., etc.) The principal mass of the oil is contained in the leaves, which yield 0.54 per. cent.

Cajeput oil as brought into commerce from the East Indies is generally contaminated with copper, by which its original green color is increased. The copper which arrives in the oil by its being dispatched in copper flasks can never be recognized by its deeper blue upon being shaken with ammonia, nor is it easily thrown down by potassium, iron, or zinc, but it is better detected by the electric current. If the poles of a voltaic battery are immersed in cajeput oil mixed with water, the wire of the positive pole becomes covered with copper oxide by the copper contained in the oil; the wire of the negative pole liberates hydrogen; upon the addition of aqueous ammonia, gas is liberated at both poles, besides that flocculi of copper form at the negative pole, and the oil becomes yellow. The dissolved copper may also be detected on shaking the oil with aqueous potassium cyanide by the red precipitate of copper cyanide. By rectification the oil becomes colorless, but resumes its green color on being left in contact with copper foil. The oil behaves in this respect like some other oils; when freshly distilled from the plants, it is colored green; on rectifying, however, it comes over colorless; oil of thyme, when distilled from the plant, is deep brown; when rectified, colorless.

The rectified oil has a peculiar strong odor, which might be compared to that of a mixture of oil of rosemary and camphor. Pure oil of cajeput is sparingly soluble in water, but freely and entirely in alcohol. It is miscible with glacial acetic acid in all proportions. Its specific gravity varies between 0.897 and 0.978 at 59° F., and it boils at from 343.5° to 347° F. It is slightly levo-rotatory.

Cajeput oil consists of about 67 per cent. cineol, $C_{10}H_{18}O$. It contains also a terpene, $C_{10}H_{16}$, which boils at 311° F., and yields a monohydrochloride melting at 260.6° to 262.4° F.; further terepineol, $C_{10}H_{18}O$, and a small amount of the various aldehydes, esters and polyterpenes.

Rectified cajeput oil converts potassium into potassa without being colored brown. It absorbs 0.35 to 0.41 per cent. caustic potash and 71 to 151 per cent. iodine, the latter being taken up without more violent reaction. Hydrochloric acid colors the oil first pale bluish red, then dirty reddish and, on heating, brownish. Sulphuric acid colors the oil brownish red, the acid becoming thereby red yellow. Alcoholic potash lye dissolves the oil with a yellow color, which, on heating, becomes brownish.

Cajeput oil is much adulterated. Formerly fictitious cajeput oil was even brought into the market, such imitation consisting of a green-colored solution of camphor in rosemary oil, or a mixture of oils of turpentine, lavender and chenopodium oils. Even at the present time the oil is adulterated with oils of turpentine and rosemary.

The behavior of cajeput oil towards iodine may serve as a guide for the detection of certain adulterations, the iodine being absorbed by the oil without detonation. With alcohol cajeput oil forms a clear solution. The oil should also burn without leaving a residue.

Cajeput oil is used in medicine as a powerful antispasmodic and diffusible stimulant.

*Cheken leaf oil* (*Chekenblätteröl*, G.) is obtained from the leaves of *Myrtus Cheken* or *Eugenia Cheken*, an evergreen shrub indigenous to Chile. The yield is about 1 per cent. The oil has a pale yellow to greenish color, and an odor somewhat between Spanish and Corsican myrrh oils. It consists of 75 per cent. pinene, $C_{10}H_{16}$, boiling between 312.8° and 314.6° F., and 25 per cent. cineol, $C_{10}H_{18}O$, boiling at 348.8° F. The oil is dextro-rotatory. Besides for medicinal purposes it is suitable for use in perfumery if it can be obtained at a reasonable cost.

*Pimento oil or oil of allspice, oleum pimentæ* (*Nelkenpfeffer— or Pimentöl*, G.; *essence de piment ou de tout-epice*, F.) The ripe fruit of *Eugenia Pimenta*, De Cand. = *Myrtus Pimenta*, L. is brought into commerce under the name pimento or allspice, and yields the better quality of pimento oil, a similar oil obtained from the leaves of the tree possessing a less delicate odor. A cheaper variety of oil is distilled in London and perhaps in India from pimento waste. The best quality of pimento is derived from Jamaica (*fructus Amoni*), Barbadoes, Cuba and other West Indian islands. Distinguished from it is, according to Piesse, the Tobasco pimento (Mexican, Spanish pimento) which is exported from Mexico, Yucatan and Cumana; it is derived from *Myrtus acris*, according to Seubert, from *Myrtus Tobasco*, W., and in less aromatic. Crown pimento is derived from *Myrtus pimentoïdes*, Nees. Finally by *piment*, cayenne pepper is frequently understood in France.

For the purpose of obtaining the oil the pimento is comminuted, or even pulverized, and distilled with steam. The distillate is at first milky turbid, but soon clarifies, an oil separating on the bottom. The yield amounts to 2.34 to 5 per cent.

Pimento oil resembles clove oil; it is colorless to slightly yellowish, smells like clove oil and has a pungent taste. It has a specific gravity of 1.04 to 1.05 at 59° F., is thickly-fluid and highly refractive.

Pimento oil, according to Oeser's investigations, consists of 67 per cent. sesquiterpene, $C_{15}H_{24}$, and 33 per cent. eugenol, $C_{10}H_{12}O_2$. However, according to Stohmann, it contains 50 to 70 per cent. eugenol and 50 to 30 per cent. sesquiterpene.

When shaken with ammonia, pimento oil, in a fresh state, remains undissolved and unchanged, but when old, is partially or entirely converted into a crystalline mass. With phloroglucin solution and hydrochloric acid pimento oil yields, according to A. Ihl, a rose color, and on boiling with resorcin becomes dirty violet. Alcoholic aniline sulphate solution colors the oil yellow on addition of dilute hydrochloric or sulphuric acid. By adding 3 drops of pimento oil to 3 drops of concentrated sulphuric acid, allowing the mixture to stand half an hour, and then adding 3 cubic centimeters alcohol, a brown resin is formed which, on boiling, dissolves with a beautiful red-brown color (G. M. Beringer).

Pimento oil may, in most cases, be used as a substitute for clove oil, but on account of its less delicate odor it is not of equal value for perfuming purposes. Pimento essence which may be recommended for cheap perfumes, is made by dissolving 3½ ozs. of pimento oil in about 5 quarts of rectified alcohol.

*Bay oil*, *oleum myrciæ* (*Bayöl*, G.; *essence de myrcie*, F.) is extracted by distillation from the leaves of *Pimenta acris*, Wight. = *Myrcia acris*, D. C., or the bayberry tree. Many varieties of the tree exist throughout the West Indies, which are scarcely to be distinguished botanically, but have quite a different odor from that of the genuine tree. Great care must, therefore, be taken in the collection of the leaves which are to be used, as the admixture of a small quantity of the other leaves may entirely spoil the product of distillation. Two oils are obtained, a "light oil" with a specific gravity of 0.870 to 0.990, and a heavy oil with a specific gravity of 1.023 to 1.037. The oil when first distilled is colorless, but by exposure to the air quickly acquires a yellowish tint, and, if the exposure be continued, becomes quite dark in color just like the oil of clove or the oil of pimento. The odor of the freshly distilled oil is rank, but in the course of from three to six months it becomes mellow, and ripens into the agreeable fragrance so much liked in the best specimens of bay-rum. The oil is soluble in 95 per cent. alcohol in all proportions, also in ether and petroleum benzene.

Bay oil equal in quality to the St. Thomas product has been obtained from the dry leaves by Schimmel & Co.

When mixed with alcoholic potash lye, bay oil solidifies to a crystalline paste; a semi-fluid mass is formed by mixing it with the same volume of a strong potash solution. By mixing in a narrow test-ube 3 drops of bay oil and 3 drops of pure concentrated sulphuric acid, and allowing the mixture to stand for half an hour until resinification is complete, the residue when heated to boiling with 3 cubic centimeters of alcohol does not color the latter, or at the utmost imparts to it a pale brown color. Oils of pimento and cloves treated in the same manner yield red colorations. Bay oil reduces ammoniacal silver solution.

Bay oil has been subjected to a careful examination by Drs. Power and Kleber. The result of their investigations has been published in the New York "Pharmaceutical Rundschau," No. 13, 1895. The following abstract of this highly interesting paper is found in the Semi-annual Report of Schimmel & Co., April, 1895:

"The only chemical examination heretofore made of this oil is that by O. Mittmann,* who obtained by the fractional distillation of the oil a relatively small amount boiling between 320° and 365° F., which he considered to consist of pinene and another terpene, probably dipentene. Mittmann also found the oil to contain a diterpene, $C_{20}H_{32}$, eugenol, and some methyl-eugenol.

"Although the investigation referred to may appear to have afforded a correct indication of the composition of the oil of bay, there were nevertheless some controverted points regarding the varying solubility of the oil, which, not being dependent upon variations in specific gravity, could in our opinion only be satisfactorily explained by its further chemical examination.

"The present U. S. Pharmacopœia (revision of 1890) states that this oil 'yields with alcohol a slightly turbid solution,' but our more recent experience with numerous distillations of this oil on a large scale has shown that the freshly distilled oil usually, if not always, forms a perfectly clear solution with alcohol of 90 per cent, and may retain this character for some time, although its solubility, even when carefully kept, gradually becomes lessened.

"The solubility of the oil is not dependent upon its specific gravity or the relative amount of phenols which it contains, as might reasonably be assumed from its accepted composition, for we have observed that some oils may afford a perfectly clear solution with alcohol and yet have a lower specific gravity than others which form a turbid solution.

"In view of these facts it was evident to us that the results of the chemical examinations of the oil of bay by Mittmann were in need of amplification or correction in several important particulars. We were thus led to take up the study of the chemical composition of this oil, and have employed for this purpose an oil of our own distillation.

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* Archiv der Pharmacie, 1889, 529.

"On fractionating a quantity of the oil it began to distil, as stated by Mittmann, at about 329° F. From 329° to 392° F. about 15 per cent. distilled over, the temperature then rose rapidly to about 473°, and up to 518° an additional 50 per cent. was obtained. From 518° to 608° F. only a few drops distilled over. A considerable amount of a thick residue remained in the flask.

"When in the course of a second fractionation, made for the purpose of purifying the fraction collected below 392° F., a high dephlegmator was used, it was shown that only about 25 per cent. of the entire liquid distilled between 329° and 347° F., then the distillation ceased completely. The residue in the flask consisted of a thick oil, which only began to distil above 572° F., with accompanying decomposition. This diterpene, therefore, was not an original constituent of the oil, but has been formed by the subsequent distillation of the latter.

"When this fact had been proved, the oil, having been first freed from phenol, was distilled in vacuo under 20 mm. pressure, whereby 80 per cent. distilled over between 152.6° and 176° F., the remaining portion distilling at 320° F.

"By repeated fractionation in vacuo we succeeded in reducing by far the greater portion of the fraction obtained between 152.6° and 176° F. to the boiling point of 152.6° to 154.4° F., under a pressure of 20 mm. This then formed a colorless, very limpid liquid, having a characteristic odor not at all like that of the known terpenes. Two analyses of this body afforded the following results:

| I. | II. | Calculated for $C_{10}H_{16}$ |
|---|---|---|
| C 88.7 per cent. | 88.2 per cent. | C 88.24 per cent. |
| H 11.4 " | 11.6 " | H 11.76 " |

"A determination of the molecular weight by Beckmann's method showed beyond doubt that the substance has the formula $C_{10}H_{16}$. Its specific gravity is 0.8023 at 59° F., its index of refraction $n_D$ 1.4673, from which 47.1 was calculated as representing the molecular refraction showing the body to possess three double bonds. But as, under such an arrangement of the atoms, a ring formation is out of the question, the atoms must form an open chain. This is borne out by the specific gravity, which is much lower than of all known terpenes. We have, therefore, in this body a new hydrocarbon, for which we propose the name of *myrcene*.

" All attempts to obtain some well-characterized addition-product of myrcene have so far been unsuccessful. Bromine is absorbed by myrcene in large amounts, but the quantity is always slightly smaller than might be expected from the three double bonds. This fact is probably due to the ready polymerization of the substance. This polymerization is, in fact, so great, that the body, which at first is of a very thinly-liquid consistency, is converted into a thick oil when exposed for a week to light and air. Myrcene is readily oxidized by permanganate, even in the cold, with the formation of some succinic acid.

"With the hope of obtaining from myrcene an alcohol of the formula $C_{10}H_{18}O$, we applied Bertram's hydrolysis method, and digested myrcene at 104° F., with glacial acetic acid, and sulphuric acid. We thus obtained an oil having a lavender-like odor, which, after saponification and subsequent fractional distillation, yielded, besides a small quantity of unaltered myrcene, dipentene (melting point of the tetrabromide 255.2° F.) and a body possessing the characteristic odor of linalool. In order to identify the latter it was oxidized, when it yielded a product which was recognized as citral by conversion into citryl-$\beta$-naphtocinchoninic acid.

" Myrcene, therefore, bears the same relation to linalool as camphene does to isoborneol, or pinene and dipentene to terpineol.

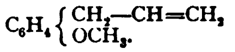
"Besides myrcene, oil of bay contains laevo-phellandrene;

but no other terpenes were found in it. Mittmann's assumption that it does contain pinene is therefore incorrect.

"The portions of the oil, free from phenols and terpenes, when shaken with a solution of sodium bisulphite, yielded a considerable amount of a solid compound, which upon being decomposed yielded a citral, identified as such in the manner described above.

"The oil remaining after the separation of the citral possessed an anise-like odor. A fraction boiling at 273.2° F. under 25 mm. pressure, which did not solidify when cooled down to —4° F., yielded anisic acid upon oxidation.

"After this fraction had been heated in a sealed tube with an alcoholic solution of potassium hydroxide to 392° F. it solidified to a white, crystalline mass, which after purification was found to be anethol, having a melting point of 69.8° F. It follows from this that besides the methyl-eugenol found by Mittmann, oil of bay also contains methyl-chavicol, or para-methoxy-allylbenzol.



$$C_6H_4\left\{\begin{array}{l}CH_2\text{—}CH{=}CH_2\\ OCH_3.\end{array}\right.$$

This body has also been found, in our laboratory, in oil of anise-bark, in estragon oil, and in oil from the leaves of *Persea gratissima*.

"Up to the present it has been assumed that eugenol was the only phenol of oil of bay. This, however, is not correct, for upon treating the crude phenol with methyliodide it yielded a mixture of methyl-eugenol and methyl-chavicol; from which it follows that besides eugenol, oil of bay also contains chavicol. Placed in order of the proportions in which they occur, the constituents of bay oil are therefore as follows:

| | |
|---|---|
| 1. Eugenol | $C_{10}H_{12}O_2$ |
| 2. Myrcene | $C_{10}H_{16}$ |
| 3. Chavicol | $C_9H_{10}O$ |
| 4. Methyl-eugenol | $C_{11}H_{14}O_2$ |
| 5. Methyl-chavicol | $C_{10}H_{12}O$ |
| 6. Phellandrene | $C_{10}H_{16}$ |
| 7. Citral | $C_{10}H_{16}O$. |

In conclusion a few points which are of practical interest may be mentioned.

"The specific gravity of the oil is naturally subject to considerable variation, being dependent upon the relative proportions of its complex constituents. With exceedingly rare exceptions it falls within the limits of 0.965 and 0.985 at 59° F., and is commonly above 0.970.

"The amount of phenols, ranging between 60 and 65 per cent., may be approximately estimated as follows. In a flask having a capacity of about 100 ccm. and provided with a long narrow neck graduated in tenths of a cubic centimeter (the same as that previously recommended by us for the examination of cassia oil), 10 ccm. of the oil are placed by means of a pipette, then 10 ccm. of a strong solution of potash are added (40 grammes KOH dissolved in water and diluted to the measure of 100 ccm.), the mixture is well agitated, and finally diluted with successive portions of water, with agitation, until the oil, being insoluble in alkali, rises in the upper portion of the neck of the flask, the graduations of which show the quantity present. The results of a few such estimations may here be recorded.

| Specific gravity of oil at 59° F. | Phenols present. |
|---|---|
| 0.962 | 59.0 per cent. |
| 0.965 | 60.5 " " |
| 0.967 | 59.0 " " |
| 0.970 | 61.5 " " |
| 0.975 | 62.5 " " |
| 0.975 | 61.5 " " |
| 0.976 | 64.0 " " |
| 0.982 | 65.0 " " |
| 0.982 | 65.0 " " |

The decrease in solubility to which we have referred is due to the conversion of the very unstable myrcene into sparingly soluble diterpene by the action of light and air.

"As we have shown that a pure oil of bay contains no pinene, this fact may be utilized, in connection with the specific gravity, for the detection of adulterations with oil of turpentine.

"If, for example, from 10 ccm. of the oil, contained in a small fractionating flask, 1 ccm. be slowly distilled off, this will contain most of the pinene which may be present. To this liquid, contained in a test-tube are added, first, 1 ccm. of amyl nitrite, then 2 ccm. of glacial acetic acid. Subsequently, while the mixture is kept cool by immersing the tube in a mixture of ice and salt, a mixture of equal parts of glacial acetic acid and concentrated hydrochloric acid is added, by drops, and with constant agitation, so long as the liquid acquires a bluish coloration. If pinene is present, a white, crystalline precipitate of pinene nitroso-chloride, $C_{10}H_{16}NOCl$, will gradually be produced. We have found that the presence of 10 per cent. of oil of turpentine in oil of bay can be readily detected by means of this reaction."

Bay oil is used as a remedy for toothache and headache, for sprinkling in sick rooms, and chiefly in the preparation of bay rum, the latter being an alcoholic solution of oils of bay, sweet orange and pimento (16: 1 : 1) in 1000 parts of alcohol and 782 parts of water.

*Myrtle oil* (*Myrtenöl*, G.; *essence de myrte*, F.). The common myrtle (*Myrtus communis*, L.) is indigenous to Southern Europe. From its leaves a very fragrant volatile oil may be obtained by distillation with water. The yield is given as 0.31 per cent. Corsican myrrh oil is considered the best, though the Spanish product is also of good quality, while the French is of less value.

Myrtle oil, according to E. Jahns, is pale yellow, has a specific gravity of 0.91 at 60.8° F., and is dextro-rotatory. It commences to boil at 320° F., and up to 464° F., 80 per cent. distils off. According to Schimmel & Co., the specific gravity ranges from 0.89 to 0.92 at 59° F. The odor of the oil is very agreeable.

According to J. H. Gladstone, myrtle oil consists of terpene, $C_{10}H_{16}$ (75 per cent.) which boils from 320° to 338° F. From the red brown residue of distillation sulphuretted hydrogen is said to evolve. According to E. Jahns, myrtle oil contains a dextro-rotatory terpene boiling between 316.4 and 320° F°., which very probably is dextro-pinene; further cyneol boiling at 348.8° F., and apparently a small quantity of a camphor, $C_{10}H_{16}O$.

Myrtle oil has become of some importance since Schimmel & Co. have brought into commerce, under the name of *myrtol*, the portion of the oil boiling between 320° and 356° F. Myrtol does good service in certain bronchial catarrhs, catarrhs of consumptives, and in affections of the bladder, and is a powerful disinfectant. Jahns designates myrtol a rectified myrtle oil, and shows it to be a mixture of dextro-pinene and cineol. Recently the best quality of myrtle oil is also used in perfumery for the preparation of specialties. In France a *myrtle flower water* (*eau d'anges*) is prepared from the flowers of the myrtle in the same manner as rose-water.

*Eucalyptus oil, oleum eucalypti* (*Eukalyptusöl*, G.; *essence d'eucalypte*, F.) is derived from various species of *Eucalyptus*. The genus, comprising some 140 known species, forms one of most characteristic features of the vegation of Australia, where the trees are known as "gum trees" or "string-bark trees." They constitute four-fifths of Australian vegetation, and are especially notable for their extraordinary fever-destroying properties. Since the introduction of the eucalyptus oil industry by Messrs. Bosisto & Co., of Melbourne, Australia, the oil has been prepared from a number of varieties which grow in Australia, Africa and California, and partially are cultivated in Italy. The species known as *Eucalyptus globulus* and *E. amgydalina* are, however, the most important. The oil is obtained by distillation with steam. Schimmel & Co. produce oil from leaves imported from Southern France and Africa. In Southern France the yield from leaves of *E. globulus* is, according to Adrain, 0.4 to 0.7 per cent. and, according to Cloëz, 2.75 per cent. from good fresh leaves. Schimmel & Co. give the yield from dried leaves of *E. globulus* as 1.6 to 3 per cent.

*Common eucalyptus oil* (from *Eucalyptus globulus*, L.) This tree is indigenous to Tasmania, but is also much grown in south-western Europe. According to S. Cloëz, fresh leaves, touched by frost, of young trees grown in Paris, yielded by distillation 2.75 per cent. of oil, while dried leaves from trees in Hyères produced 6 per cent., and leaves derived from trees in Melbourne, which had been kept for five years, only 1.5 per cent. The oil was limpid, almost colorless, and possessed a camphor-like odor. It commenced to boil at 338° F.; the thermometer then rose to 347° F., and remained at this temperature until about one-half of the oil had been distilled off. Another portion passed over at 370.4° to 374° F., and a small quantity above 392° F. The specific gravity of oil from *E. globulus* is, according to Jahns, 0.921 at 59° F.; according to Schimmel & Co., it ranges from 0.91 to 0.93 at 59° F., and the optical rotation is + 1° to + 20°.

The known constituents of eucalyptus oil from *E. globulus* are 50 to 70 per cent. cineol (eucalyptol), $C_{10}H_{18}O$; further dextro-pinene $C_{10}H_{16}$, and small quantities of valeraldehyde, butyraldehyde and capronaldehyde. According to Williams, the oil dissolves 61.68 to 110.65 per cent. of iodine without detonation.

Eucalyptus oil from *E. globulus* is chiefly used for medicinal purposes, but is also employed in perfumery. Most of the oil comes from Algiers and California.

*Australian eucalyptus oil*, from *Eucalyptus amygdalina*, L., is limpid, pale yellow, possesses an odor reminding one somewhat of lemons, and a taste at first mild and cooling, later on bitter. Its specific gravity ranges from 0.86 to 0.96; optical rotation —27° to —68°. It boils between 329° and 370.4° F. On exposure to the air it resinifies and at —0.4° F. separates a stearoptene which melts again at 26.6° F. (Stohmann.)

The principal known constituents of Australian eucalyptus oil are cineol and phellandrene. It appears, however, to be poorer in cineol than the oil derived from *E. globulus*. It is used for perfuming soaps, and for diluting the more delicate oils used in perfumery.

*Other eucalyptus oils*. The following list of eucalyptus oils of different derivation is compiled from statements by F. v.

Müller and Bosisto, and from the reports of Messrs. Schimmel & Co.

*Eucalyptus oleosa:* Thin, pale yellow oil of a camphor-like odor and peppermint-like taste. Specific gravity 0.915 to 0.925 at 59° F.; boiling point 300.2° to 350.6° F.; optical rotation —3° and —5°; known constituents: cineol and cuminol.

*Eucalyptus sideroxylon:* Pale yellow oil resembling the preceding. Specific gravity 0.918; boiling point 305.6° to 347° F.

*Eucalyptus corymbosa:* Colorless oil possessing an odor of roses and lemons. Specific gravity 0.881.

*Eucalyptus obliqua:* Reddish yellow oil of a mild odor; boiling point 339.8° to 375.8; specific gravity 0.903.

*Eucalyptus odorata:* Pale greenish yellow oil; specific gravity 0.907 at 59° F.; principal constituents: cineol and cuminol. According to Schimmel & Co., this oil is cheaper than the product from *E. amygdalina,* and at the same time richer in cineol, so that it may be used for obtaining the latter. Generally speaking it resembles Australian eucalyptus oil.

*Eucalyptus longifolia:* Thickish oil with a strong odor of camphor. Specific gravity 0.94; boiling point 381.2° to 419° F.

*Eucalyptus rostrata:* Pale yellow oil distinguished by its low boiling point of 267.8° to 357.8° F. Specific gravity 0.924 at 59° F., optical rotation +12° 58′; known constituents: valeraldehyde, cineol.

*Eucalyptus viminalis:* An oil of a very disagreeable odor, boiling between 318.2° and 359.6° F., and having a specific gravity of 0.918 to 0.921.

*Eucalyptus resinifera.* From this plant the New Holland kino is derived. The oil consists chiefly of a hydrocarbon smelling of oil of turpentine.

*Eucalyptus Baleyana:* Specific gravity of the oil 0.940 at 59° F.; boils between 320° and 365° F.; contains about 30 per cent. cineol and 70 per cent. terpene, probably dextropinene.

*Eucalyptus microcorys:* Specific gravity 0.935; boiling point between 320° and 392° F.; composition same as the preceding.

*Eucalyptus dumosa:* The oil is very rich in cineol.

*Eucalyptus dealbata:* Specific gravity of the oil from dried leaves 0.946 at 59° F.; from fresh leaves 0.885. Yield from dried leaves 2.7 per cent. Boiling point between 402.8° and 420.8° F. The oil has an agreeable balm-like odor; it is free from terpene and contains a ketone, $C_{10}H_{16}O$, of a balm-like odor, and an alcohol ($C_{10}H_{18}O$?) smelling of geranium.

*Eucalyptus maculata:* Specific gravity 0.90 at 59° F.; boils between 410° and 428° F.; otherwise resembles the preceding oil.

*Eucalyptus maculata var. citriodora*, Hérit. (*lemon-scented gum-tree oil, essence de gommier à odeur de citron*, F.; *Schönminzöl*, G.) By rubbing between the fingers, the leaves of this plant evolve an agreeable odor reminding one of lemon, balm and citronella, the dried leaves imparting this odor to clothes when placed between them. The oil was first obtained by Norie from the leaves by distillation with water. The yield was 0.7 to 1.7 per cent. The oil is almost colorless; specific gravity 0.87 to 0.905 at 59° F. It is slightly dextro-rotatory, boils between 408.2° and 428° F., and contains as known constituents citronellon and geraniol. The yield from dried leaves is 2.2 per cent.

*Eucalyptus Stageriana:* Specific gravity, 0.88 at 59° F.; boils between 433.4° and 451.4° F., possesses an intense odor of lemon and verbena, and contains a ketone ($C_{10}H_{16}O$?) of a strong pure lemon odor, and in addition considerable quantities of a terpene. Yield, 3.7 per cent.

*Eucalyptus Backhausia = Backhausia citriodora:* Specific gravity, 0.9 at 59° F.; boils between 433.4° and 451.4° F. The oil resembles the preceding, but contains only a little terpene. According to Schimmel & Co., the ketone-like body (boiling point between 431.6° and 440.6° F.) is difficult to purify, since it does not distil undecomposed and very readily resinifies. It appears to have the formula $C_{10}H_{16}O$; it is called citral, possesses an intense lemon odor, and is said to occur (6 to 8 per cent.) in lemon oil, in large quantities in lemon grass oil and up to 30 per cent. in citronella oil.

*Eucalyptus Haemastoma:* Smells like cumin oil; specific gravity, 0.89 at 59° F.: boils between 338° and 482° F. It contains as known constituents cymol, menthone (?), cuminaldehyde.

*Note.* From the oils of *Eucalyptus globulus* and *E. odorata, eucalyptol or cineol* is prepared on a large scale and much used as a substitute for the oil. It is a powerful antiseptic agent, has a preserving effect, and in a solution of 1 : 166 prevents the formation of bacteria. It is used in subcutaneous injections in phthisis, and is also suitable for the preparation of cosmetics, as well as for perfuming soaps.

From Australia impure eucalyptol with a specific gravity of 0.92 at 59° F., and boiling between 347° and 352.4° F. is brought into commerce; it is, however, of little value compared with the pure product. Pure eucalyptol has a specific gravity of 0.93 at 59° F., boils between 348.8° and 350.6° F., is optically inactive, and in a freezing mixture solidifies to long colorless needles.*

Mr. Scammel, of Adelaide, has recently taken out a patent for the preparation of eucalyptol from eucalyptus oil and other volatile oils. His process is based upon the hitherto unknown property of eucalyptol to from a crystalline compound with phosphoric acid, from which the eucalyptol may be separated again by means of water. The rationale of this process is therefore the same as that of the well-known addition-products of hydrochloric acid and hydrobromic acid.

The process consists in mixing oil of eucalyptus with slightly more than the necessary quantity of phosphoric acid of specific gravity 1.8, with constant stirring, care being taken to cool the mixture well. The eucalyptol-phosphate which crystallizes out is then purified by pressing and afterwards decomposed by means of hot water.

*Clove oil, oleum caryophylli (Nelkenöl,* G.; *essence de Girofle,* F.) Every part of *Eugenia caryophyllata,* Thunb. = *Caryophyllus aromaticus,* L., or the clove tree, abounds with aromatic oil, but it is most fragrant and plentiful in the unexpanded flower-buds, which are the cloves of commerce. They are soaked for some time in salt-water and then submitted to distillation in a still, generally of boiler iron. The distillate is received into a tall tinned vessel furnished with a funnel at the top projecting down towards its centre, and from near the top a lateral tube issues, bent upwards and then downwards so as to carry off the distilled water which collects above the oil. As the distilled water accumulates, it is from time to time returned into the still. The yield of oil is about 16 per cent.

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* Schimmel & Co., Report, April, 1890.

An attempt by Ender to obtain the oil of cloves by percolating powdered cloves with carbon disulphide, displacing the latter by water and distilling the percolate, previously separated from the aqueous layer, proved a failure; the oil possessed a peculiar unpleasant odor, due to a little of the disulphide, which made it unfit for use.

Clove oil, when fresh, is yellowish and thickly-fluid, but by age becomes yellow to brown. It has the aromatic taste and odor of cloves. Its specific gravity ranges from 1.060 to 1.070 at 59° F.; it is slightly levo-rotatory, and, according to Williams, boils at 476.6° F. It may be cooled to 0.4° F. without solidifying. It is sparingly soluble in water, but very readily in alcohol, ether, glacial acetic acid and fat oils. It has, as a rule, a slightly acid reaction.

Clove oil consists of eugenol, $C_{10}H_{12}O_2$ (90 per cent.) sesquiterpene, $C_{15}H_{24}$ (10 per cent.), and traces of vanillin.

V. Coblentz has investigated the action of clove oil upon metals, and found that lead, tin and bismuth are not attacked by it, iron very slightly, but zinc and copper very vigorously. With zinc, clove oil forms a gray fatty mass from which separates a colorless oil possessing a slight clove-odor. In copper vessels the oil turns red-brown, and separates a sediment of the same color. In iron vessels the oil becomes decolorized and separates a slight, gray sediment. Iodine, according to Williams to the extent of 155 to 180 per cent., dissolves slowly in clove oil without a perceptible increase in the temperature. Chlorine thickens the oil and colors it green. Caustic potash is dissolved by the oil to the extent of 2.4 to 2.5 per cent. Potash lye, soda lye and ammonia convert clove oil into a crystalline paste. By shaking 3 drops of clove oil with 3 drops of pure concentrated sulphuric acid, and allowing the mixture to stand for half an hour, a purple resin is formed which, by shaking with 3 cubic centimemeters of 50 per cent. alcohol and heating to boiling, dissolves with a red color. The solution acquires a purple fluorescence (Beringer). With two drops of sulphuric acid and 6 drops of oil, the acid is colored reddish, the oil brown, bluish, violet, indigo, and finally pure Prussian blue. Fuming nitric acid ignites the oil, while ordinary nitric acid oxidizes it on heating to resinous masses and oxalic acid. Alcoholic phloroglucin solution + hydrochloric acid colors the oil pale and fiery red; resorcin, on boiling, colors it red violet, and pyrogallic acid, on boiling, violet.

Clove oil is frequently adulterated with oils of copaiva and cedar wood, almond and castor, with alcoholic extract of cloves and colophony solution, with clove-stem oil, alcohol, oils of turpentine and pimento, oils of sassafras, thyme and rosemary, and finally even with carbolic acid. Sophistication with the latter may be detected by Flückiger's test as follows: Agitate the suspected oil with 50 parts of hot water; decant and slowly evaporate the aqueous portion to a small bulk. Add one drop of aqua ammonia and a very little chlorinated lime. If carbolic acid is present, a green color, changing to a permanent blue, is developed.

Generally speaking, pure clove oil should possess a pure clove-odor and entirely dissolve in alcohol or ether. Upon paper it should not leave behind a permanent grease stain. To water it should not yield perceptible quantities of soluble matters, nor separate an oil specifically lighter than water. Finally, when shaken with potash lye, it should form a clear mixture, and after standing for 3 hours solidify to a crystalline mass without oily drops being separated on the surface after standing for some time.

Clove oil is extensively used especially in the perfumery industry. A solution of 3½ ozs. of it in 4 quarts of alcohol forms *clove essence.* The therapeutic action and uses of clove oil are essentially the same as those of the oils of caraway and anise. It is seldom employed internally, but is sometimes used to allay the pain of carious teeth and of earache. Finally, from clove oil is obtained eugenol, which may serve for the preparation of vanillin.

*Clove stalk oil* (*Nelkenstielöl,* G; *essence de griffes ou queues de girofle,* F.) is obtained by distilling the stems and waste of cloves. The oil is greenish yellow, generally darker red-brown than older clove oil, possesses a clove-odor with a disagreeable, herb-like by-odor, and the taste of clove oil. It dissolves with greater difficulty than clove oil, and, as a rule, has not an acid reaction. Its specific gravity ranges from 1.55 to 1.65 at 59° F. It is said to be poorer in eugenol than clove oil, and richer in sesquiterpene.

### *Rosaceæ.*

*Rose oil, ater or attar of roses, incorrectly also otto of roses, oleum rosarum,* (*Rosenöl,* G.; *essence de rosa,* F.) Under this name various products are brought into commerce, some of them being actually derived from different species of roses, while others are rose oils only in name.

The profitable cultivation of roses for the preparation of oil is limited chiefly by climatic conditions. The odoriferous constituent of the oil is a liquid containing oxygen, the solid hydrocarbon of stearoptene with which it is combined being absolutely devoid of perfume. The proportion which this inodorous solid constituent bears to the liquid perfume increases with the unsuitability of the climate, varying from about 18 per cent. in Bulgarian oil to 35 and even 68 per cent. in rose oils distilled in France and in England. This increase in the proportion of stearoptene is also shown by the progressively heightened fusing points of rose oils from different sources; thus, while Bulgarian oil fuses at about 61° to 64° F., an Indian sample required 68° F., one from the south of France 70° to 73° F., one from Paris 84° F., and one obtained in making rose-water in London 86° to 89½° F.

The cultivation of roses for the preparation of oil is carried on to a considerable extent in Persia and India, a large area being devoted to rose gardens in the latter country. The species of roses used is *Rosa damascena.* The flowers are distilled with double their weight of water in clay stills; the rose water thus obtained is placed in shallow vessels, covered with moist muslin to keep out dust and flies, and exposed all night to the cool air, or fanned. In the morning the film of oil which has collected on top is skimmed off by a feather and transferred to a small phial. This is repeated for several nights, till almost the whole of the oil has separated. The quantity of the product varies much, and the color ranges from green to bright amber and reddish. The oil is most carefully bottled, the receptacles are hermetically sealed with wax and exposed to the full glare of the sun for several days.

Some rose oil is produced in Tunis, the rose grown for the purpose being the dog rose, *Rosa canina.* It is extremely fragrant; 20 pounds of the flowers yield about 1 drachma of oil. Very little of this oil reaches the European market.

In the south of France rose gardens occupy a large share of attention about Grasse, Cannes, and Nice, but the oil produced is at once used in the preparation of perfumery, very little of it being brought into the market. The variety of rose chiefly cultivated is the *Rosa provincialis* or *R. centifolia.* The oil has a characteristic odor which, according to Piesse, is due to bees conveying the pollen of orange flowers to the rose-buds. It is generally said that French oil contains more stearoptene than the Bulgarian product, which is however denied by H. Massignon. At Cannes and Grasse the roses are chiefly subjected to maceration, whereby rose-pomade is obtained. Ten pounds of rose leaves suffice for the *enfleurage* of ½ lb. of fat. The best quality of French rose oil is known as *Essence de rose indigene.*

In England roses are cultivated on a large scale at Mitcham, but chiefly for the production of rose water. Towards the end of June or commencement of July the roses are gathered immediately after the dew is dried off, packed in bags and sent to London. After their arrival there, they are immediately spread out in a cool place, otherwise fermentation would set in, and the leaves become useless in 2 or 3 hours.

The Greek government has for some time been occupied with the question of introducing the cultivation of the rose in Greece and establishing the rose oil industry. The Russian government also has recently taken steps to introduce the rose oil industry in the Caucasian government Kutais. The experiments made in the Baku government have been quite successful. The Crimea is also well adapted for this purpose, especially as *Rosa centifolia* is said to grow wild in the mountains.

In the United States the cultivation of roses for the production of oil has been attempted in California, but with what success is not known.

Rose oil from roses grown in Germany was for the first time produced in 1884, by Schimmel & Co., of Leipsic. Encouraged by their success, they induced several gardeners and farmers to plant roses by the acre. The oil was of a better quality than Turkish rose oil, it possessing a more powerful and a finer odor. 100 lbs. of fresh rose leaves yield about 1 lb. of oil. To be sure the climate of Leipsic is not so favorable to the growth of roses as that of the Balkan, but the rose grown there is equal in fragrance to the Bulgarian. The variety *Rosa centifolia major* has proved especially suitable for cultivation, but being difficult to propagate, experiments on a large scale have been made with the Thracian rose. The latter is hardy and grows very rapidly; to thrive well it requires a light, calcareous, clay soil, poor in phosphoric acid, with a moderate content of nitrogen. In the fall of 1889 a strip of land in the immediate neighborhood of Leipsic was planted with 250,000 rose-stocks, so that the cultivation of roses for the production of oil in Germany may be considered securely established.

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In countries where the yield of roses is not sufficiently large to warrant the distillation of each day's crop, the flowers are pickled for preservation. For this purpose the leaves are separated from the stalks, packed with alternate layers of salt in a vat, and the whole thoroughly stirred together. For about 6 pounds of rose leaves 1 pound of common salt is required. The salt absorbs the water contained in the petals, reducing the whole to a pasty mass, which will keep for any length of time without the fragrance being seriously injured. When a sufficient quantity of this pasty mass has been accummulated, it is subjected to distillation with water in the proportion of about 25 pounds of pickled roses to 5 gallons of water. By drawing off about 4 gallons an excellent product—the so-called double-distilled rose-water of the shops—is obtained.

Commercially the most important source of rose oil is a circumscribed patch of ancient Thrace or modern Bulgaria, stretching along the southern slopes of the Central Balkan, and included between the 25th and 26th degrees of eastern longitude, and the 42d and 43d degrees of north latitude. The geological formation throughout the district is syenite, the decomposition of which has provided a soil so fertile as to need but little manuring. The rose bushes flourish best and live longest in sandy, sun-exposed slopes, with a south and south-east aspect. According to a report by Mr. Ernst Schmalfuss, an expert, who was sent by a German firm to investigate the oil-of-rose industry in Bulgaria, the variety used for distilling purposes is the so-called Thracian rose, a plant of exceedingly rapid growth, flowering sparingly in the first year, and yielding a full crop in the third, when it attains maturity. It is said that, under certain conditions, the plants attain an age of 50 years. The plant bears red or white flowers, the former being about five times as numerous as the latter. Both varieties are of a very powerful and agreeable odor, but the oil distilled from the white flowers is the finest, though the red roses are richer in essential oil. The Thracian rose exceeds all other varieties in flowering property, weak specimens bearing as many as 500 flowers, while fine plants, if properly cultivated, are able to produce double that number. The roses are grown in fields, where they are placed in rows about 2 yards apart, alternating with rows of grape-vines or kitchen vegetables. To a practical man it would appear that in the Bulgarian fields the plants are grown too closely together, and have no room left to expand properly. As a hint to intending experimenters in Western Europe, which applies also to this country, in many parts of which rose culture could, no doubt, be carried on profitably, Mr. Schmalfuss recommends that the soil be well manured with old, partly-decomposed manure, the application of which should be repeated every third year. The plants should be placed in rows, about 8000 trees to the acre, and during the first two years the rows of rose plants may alternate with rows of kitchen vegetables. It may be found to pay to cut the shrubs in the second year close to the ground. The yield of that year is, of course, lost by this proceeding, but the luxuriance of the plant for the future is thereby much increased. After the third year the planting of vegetables must be discontinued. The soil must be kept free from weeds and rendered loose twice a year by hoeing. The fields might, experimentally, be protected at the north side by hedges. The flowers must be gathered early in the morning, and placed in open baskets, which should be kept in the shade. As long as the Thracian roses are not obtainable in quantities, the export of rose-plants being prohibited by the Bulgarian government, Mr. Schmalfuss recommends the Moss, Bourbon, and Remontant roses as being richest in oil.

Almost every small Bulgarian farmer distils his own oil, the stills being of the most elementary description, and a better oil and a greater yield could, no doubt, be obtained by improved appliances. The apparatus in general use in Bulgaria consists of a tinned-copper still, erected on a semicircle of bricks, and heated by a wood fire; from the top passes a straight tin-pipe, which obliquely traverses a tube kept constantly filled with cold water, by a spout from some convenient rivulet, and constitutes the condenser. Several such stills are usually placed together, often beneath the shade of a large tree. The still is charged with 25 to 50 pounds of fresh roses, not previously deprived of their calyxes, and double the volume of spring-water. Distillation is carried on for about 1½ hours, the product being simply a very oily rose-water. The exhausted flowers are removed from the still, and the decoction is used for the next distillation instead of fresh water. The first distillates from each apparatus are mixed and distilled by themselves, ⅙ being drawn off; the residue replaces spring-water for subsequent operations. The distillate is received in long-necked bottles holding about 1¼ gallons. It is kept in them for a day or two, at a temperature not exceeding 59° F., by which time most of the oil, fluid and bright, will have reached the surface. It is skimmed off by a small, long-handled, fine-orificed tin funnel, and is then ready for sale. The last run of rose-water is extremely fragrant, and is much prized locally for culinary and medicinal purposes. The quantity and quality of the oil are much influenced by the character of the water used in distilling. When hard spring-water is used, the oil is rich in stearoptene, but less transparent and fragrant. The average quantity of product is estimated by Baur at 0.037 to 0.040 per cent.; another authority says that 3200 pounds of roses give 1 pound of oil.

Pure oil of roses, carefully distilled, is at first colorless, but quickly becomes yellowish; its specific gravity is 0.870 at 72.5° F.; its boiling-point is 444° F.; it solidifies at 52° to 61° F., or still higher; it is soluble in absolute alcohol and in acetic acid. The most usual and reliable tests of the quality of rose-oil are: 1, its odor; 2, its congealing point; and 3, its crystallization. The odor can be judged only after long experience. That of the concentrated oil is intense, penetrating, and diffusive, and to most persons unpleasant, the fine and agreeable odor being only brought out by dilution. A good oil should solidify in 5 minutes at a temperature of 54.5° F.; fraudulent additions lower the solidifying point. The crystals of rose-stearoptene are light, feathery, shining plates, filling the whole liquid.

Rose oil consists of a mixture of paraffin-like hydrocarbons (stearoptene) and geraniol (elæoptene). The stearoptene is of the most interest because it is generally believed that the more a rose-oil contains of it, *i. e.*, the more readily it solidifies, the better it is. This is true to a certain extent, in as far as adulterating with foreign oils reduces the solidifying point; but this may be equalized by the addition of solid substances, and the odor of rose oil is solely due to the liquid portion. The content of stearoptene in various oils has frequently been determined. Flückiger found in unquestionably genuine oil of Schimmel & Co., 28.86 per cent. of stearoptene, and in Turkish oil, claimed to be pure, only 9.2 per cent.. Schimmel & Co. themselves state, in 1889, that their oil contains 32.5 to 34 per cent. of stearoptene, and Turkish oil 12 to 14 per cent. The separation of the stearoptene is best effected by heating 50 grammes of the oil with 500 grammes of alcohol to between 158° and 176°F., and then cooling to 32°F., whereby the stearoptene separates almost quantitatively. The latter is then again treated in the same manner with 200 grammes of alcohol, whereby, as a rule, it is obtained pure.

The elæoptene of rose oil is brought into commerce by Schimmel & Co., as liquid rose oil or rose oil free from stearoptene. It has to be carefully protected from light and heat.

Rose oil dissolves iodine, it gradually resinifying thereby. By sulphuric acid it is dissolved without reaction, and by nitric acid with foaming. From the acid solutions water separates a fatty or waxy substance.

It is evident that such a valuable article as rose oil is very much exposed to adulteration. Indeed, it is said that only adulterated oil reaches the market, the sophistication, chiefly with geranium and ginger-grass oil, taking place at the home of the oil. The addition is generally made by sprinkling ginger-grass oil upon the rose leaves before distilling. The general characteristics of this oil (see Vol. 2, p. 375) are so similar to those of rose oil, even the odor bearing a distant resemblance, that detection is very difficult. Greiner recommends the following as a reliable test: Put one drop of the suspected oil in a dry test-tube and add four drops of concentrated sulphuric acid. A perceptible rise of temperature takes place, and the mixture must be allowed to stand until it becomes cool. Two grammes of absolute alcohol are then to be added and the mixture well shaken. When the oil is pure the mixture will be slightly opalescent, and, on heating, will turn yellowish-brown, the color remaining on cooling the solution. When the oil has been mixed with ginger grass, or geranium oil, the solution will be turbid and an insoluble preciptate soon forms. Pure rose oil retains its characteristic odor when subjected to this test, but the mixture with the other oils evolves the unpleasant odors.

When fatty oils, such as sesamé, almond, etc., are used as adulterants, the usual test, made by placing a drop on white paper and heating over an alcohol flame, shows their presence in the greasy stain which remains. Pure rose oil is entirely volatile.

The presence of spermaceti, which is usually used for artificially heightening the apparent proportion of stearoptene, is easily recognizable from its liability to settle down in a solid cake and from its melting at 122° F., whereas the stearoptene fuses at 81.5° F.

Hoppe recommends the following test to detect spermaceti: The suspected oil is agitated with one and a half to twice its weight of melted glacial acetic acid, the resulting crystalline mass transferred to a filter and washed with vinegar to remove the oil, then with water until the odor of roses has quite disappeared, and finally washed with a solution of soda and again with water. Should any residue result, its identity with spermaceti is evidenced by the odor produced, similar to burning oil when charred, and may be reproduced after some time by again heating the substance.

The specific gravity, freezing point and rotatory power of rose oil vary so greatly, according to the different localities of production and the climatic conditions which have prevailed, as to render the estimation of the oil extremely difficult.

Jedermann has published in the Zeitschrift für analytische Chemie, 1895, 51, a contribution on the testing of rose oil for geranium oil, and arrives at the following conclusion: "Up to the present we have no characteristic test by which we are able to detect with certainty the presence of geranium oil in rose oil. Consumers have to rely upon their own practical knowledge, and to take proper care to select a trustworthy source of supply." Oil of roses is put up in Bulgaria in squat-shaped flasks, holding from one to ten pounds, and sewn up in white woolen cloths. Usually their contents are transferred at Constantinople into small gilded bottles for export. The value of an average harvest is from $3,500,000 to $4,500,000.

Rose oil is chiefly used in perfumery. Medicinally it is not employed except, perhaps, for perfuming some pharmaceutical preparations. Rose water and rose essence also are frequently used. *Rose water* is obtained as a by-product in the distillation of roses with water, but is also prepared as chief product in a similar manner. A product of inferior quality is obtained by agitating water with rose oil. *Rose essence* is the alcoholic extract of rose pomades, inferior qualities being prepared by dissolving rose oil in alcohol. Six kinds of rose essences are known in commerce: *Extract de roses triple*, *de roses blanches*, *de roses-thé*, *de roses moussues*, *de rose double* and *de rose de Chine*. Rose pomade or rose oil is exclusively used only for triple essence (1 quart rectified alcohol + 1 pound of pomade; or 1 quart of alcohol + 12½ drachms rose oil); in all other cases essences or volatile oils are added for the production of the odor.

*Spiræa oil or meadow-sweet oil* (*Spiræaöl or Spierstaudenöl*, G.), is obtained by distillation with water or steam from the herb of the meadow-sweet, *Spiræa Ulmaria*, L., indigenous to Europe, and cultivated in gardens. The oil consists of salicylaldehyde, $C_6H_4(OH)COH = C_7H_6O_2$, a terpene, $C_{10}H_{16}$, and a crystalline, camphor-like body. The salicylaldehyde may be dissolved with potash lye and again separated, or may be fixed on acid alkaline sulphates, and thus obtained. The oil is very fragrant, having an aromatic and at the same time slightly bitter almond-like odor, and a pungent taste. It is quite soluble in water. It is rather expensive, and for that reason is scarcely used. It may be remarked that salicylaldehyde (a liquid) is readily converted by oxidation into the crystallizing oxybenzoic or salicylic acid, $C_6H_4OHCOOH$.

### *Drupaceæ.*

*Bitter almond oil, oleum amygdalarum amarum* (*Bittermandelöl*, G,; *essence d'amandes amères*, F.) is obtained by submitting bitter almond-cake (left after the expression of the fixed oil from bitter almonds) to distillation with water. The volatile oil does not exist ready-formed in the bitter almond, nor in the almond-cake, but results from the decomposition of a glucoside called "amygdalin" contained in the cake, under the influence of emulsin and water, the emulsin acting as a ferment, into benzaldehyde, glucose and prussic acid.

The reaction is illustrated by the following equation:—

$$\underset{\text{Amygdalin.}}{2C_{20}H_{27}NO_{11}} = \underset{\text{Benzaldehyde.}}{4C_7H_6O} + \underset{\text{Hydrocyanic acid.}}{2HCN} + \underset{\text{Glucose.}}{C_6H_{12}O_6} + \underset{\text{Formic acid}}{4CH_2O_2} + \underset{\text{Water.}}{4H_2O}$$

Benzaldehyde, which is the principal constituent of the oil, readily absords oxygen, and is thus converted into benzoic acid, $C_7H_6O + O = C_7H_6O_2$. This change takes place by exposure of the oil to the atmosphere, and hence a small quantity of benzoic acid is generally present in the commercial oil.

The oil is prepared in the following manner: The press cakes of bitter almonds are ground, then soaked about twenty-four hours in twice their weight of water, to which one-third of their weight of salt has been added. The whole is then submitted to distillation. In distilling, the hydrocyanic or prussic acid evaporates, together with the crude oil. As the acid is the most poisonous substance known, special care must be exercised to prevent any vapors from escaping into the workroom. This is best effected by hermetically fixing by means of a cork the lower end of the cooling pipe into a large bottle, in which the oil and water can condense; in the cork is also fitted a glass tube leading to a fire-place, for instance, of a boiler. The non-condensed vapors of prussic acid escape through this tube into the fire-place and are rendered innocuous by combustion.

Almond oil being readily soluble in water distilling with indirect steam is absolutely necessary, as direct distillation with water would yield aromatized water, but no oil.

The oil in its crude state contains nearly 13 per cent. of anhydrous hydrocyanic acid, and is therefore exceedingly poisonous, unless completely deprived of this substance. It can be freed from a large portion of the acid by shaking with lime, but completely so only by the following process: Mix a solution of iron in a mixture of 5 parts of hydrochloric acid and 1 part of nitric acid with the oil, add some lime-water, and stir thoroughly. After resting for some time, the mixture is rectified in the usual manner, and is thus obtained free from every trace of prussic acid.

Oil of bitter almonds is chemically the aldehyde of benzol, hence called benzylic aldehyde or benzaldehyde. An oil closely resembling it is obtained by distillation from cherry laurel leaves, the kernels of peaches, cherries, apricots, or any vegetable products containing amygdalin and emulsin. It may be prepared artificially in many ways, not only from benzol, but even from toluol by reduction. The artificial oil is identical with the true oil; so far but little of it is manufactured, the product obtained from bitter almonds being as cheap.

Bitter almond oil in a crude state is yellowish, but when rectified forms a colorless highly refractive liquid, which on exposure to the air becomes thickly-fluid, acquires a yellow color and by reason of absorbing oxygen, separates crystals (of benzoic acid, $C_6H_5COOH$). The oil has a strong odor of bitter almonds and an aromatic, agreeable, somewhat pungent taste. The specific gravity of pure benzaldehyde is 1.0636 at 32° F., or 1.0504 at 59° F.; it boils at 354.2° F. On the other hand, the specific gravity of bitter almond oil ranges from 1.036 to 1.072 at 59° F., crude oil being heavier than rectified, and fresh oil lighter than old. The generally accepted specific gravity of the oil is 1.043; it boils at 356° F. Bitter almond oil is optically inactive and when chemically pure is neutral, while old oil or oil containing prussic acid shows an acid reaction. It dissolves in 30 parts of water and readily in alcohol and ether. When heated in the air it ignites and burns with a bright and sooty flame.

The tendecy to decomposition of bitter almond oil (especially of that freed from prussic acid) is highly objectionable to consumers of the article. This property shows itself by the separation of crystals, which are particularly prone to form themselves freely and rapidly when a small quantity of oil has been allowed to remain in the bottle. This peculiarity is due to the affinity of bitter almond oil or benzaldehyde for atmospheric oxygen, with which it combines resulting in the formation of benzoic acid. The oxidation first shows itself by the sides of the upper portion of a half-filled bottle, which the benzaldehyde has touched, becoming covered with a white crystalline coating. Crystals next appear upon the surface of the liquid, sinking to the bottom as soon as they have reached a certain size and gradually penetrating the whole of the liquid, which is ultimately converted into a solid mass of benzoic acid. As the acid is inodorous and of no value to the perfumer or cordial-maker, this oxidizing process often entails a distinct loss to the consumer. As the access of air is a condition which must necessarily precede the decomposition process, it follows that, if the oil can be kept protected from the air it will not be subject to this form of deterioration. Hence, if bitter almond oil be kept in bottles filled to the neck and carefully closed, no loss from oxidation need be feared.

In practice, however, this cannot always be carried out, and in consequence of repeated enquiries for a preservative for bitter almond oil* Schimmel & Co. have made experiments to ascertain whether the evil might be prevented by the addition of alcohol to the oil, and what would be the most suitable proportion in which to add the alcohol.

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* Semi-annual Report, April, 1895.

For this purpose three bottles of equal size, each half-filled and corked, were left standing for seven weeks next to one another, *i. e.*, under the same conditions.

Bottle No. 1 contained artificial bitter almond oil, without addition.
" " 2 " " " " " with 5 per cent. of alcohol added.
" " 3 " " " " " with 10 per cent. of alcohol added.

In the course of the experiment differences were observed in the bottles. After a short time No. 1 showed crystals on the upper portion of the glass. The same formation, but in a much greater degree, was observed in No. 2, and at the close of the experiment the liquid was honeycombed with crystals. No. 3 showed no visible alteration whatever in the course of the experiment.

A quantitative determination of the free benzoic acid formed gave the following result:

| Found present in | |
|---|---|
| No. 1 (pure benzaldehyde) | 12.8 per cent. benzoic acid. |
| " 2 (5 per cent. of added alcohol) | 31.4 " " " " |
| " 3 (10 " " " " " ) | 1.3 " " " " |

This experiment shows beyond doubt that the addition of 10 per cent. of alcohol has a strong preserving influence on bitter almond oil, whereas a smaller addition not only fails to act as a preservative, but is directly injurious to the oil.

It is, therefore, advisable either to keep bitter almond oil in well-corked bottles, filled to the neck, or where that is impossible, to add 10 per cent. of alcohol to the oil. It follows that in the latter case 11 parts of the mixture should be given where 10 parts of the pure oil would be dispensed.

Experience has shown that bitter almond deprived of prussic acic is much more liable to oxidation than oil containing prussic acid. The prussic acid, therefore, has the same preserving action as the addition of a considerable percentage of alcohol.

As an example of the avidity with which bitter almond oil absorbs oxygen when brought in contact with atmospheric air, it may be mentioned, that when 2 grammes of benzaldehyde were left standing in an open beaker overnight, it was found next morning to contain 20.7 per cent. of freee benzoic acid.

Oil of bitter almonds is much adulterated with cheaper oils, but chiefly with nitrobenzole, which has an odor very similar to that of the genuine oil. Pure almond oil mixed with *sulphuric acid* gives a clear crimson-red color without perceptible decomposition; with an *alcoholic solution of potash*, crystals are eliminated. The oil will take up as much as one-third of its weight of *iodine* and retain the same in solution. *Potassium chromate* does not affect the oil. To detect nitrobenzole, Dr. E. Pegna recommends the following method as uniting convenience with accuracy: The suspected oil is treated with a small quantity of alcohol and solution of potassic hydrate (purified by alcohol), and a few drops of ferric chloride are then added. The mixture is allowed to stand several hours, is then shaken and distilled. A portion of the oil collected in the receiver, after being deprived of water by suitable means, is then poured into a test-tube containing a few pieces of pure potassic hydrate, and heated over a lamp. If the oil is pure it remains colorless, because the hydrate of benzol is converted into benzoate of potassium and hydrogen. If, on the contrary, it contains nitrobenzole, a dark color is produced, owing to the formation of nitrobenzid; and since by this reaction a small quantity of aniline is also produced, a few drops of solution of chlorinated lime poured upon the cooled mixture acquire a violet color. In conducting this process it is necessary to avoid all bumping and spirting, so that the distillate may not become contaminated with portions not properly belonging to it. It is to be observed also that no portion of the oil is exposed to the direct flame during the distillation, which should proceed with the utmost regularity.

According to Bourgoin, the presence of nitrobenzole is detected by shaking the suspected oil in a test-tube with one-half its weight of solid caustic potash. The yellow color of the oil is not changed if it is pure, but if nitrobenzole is present the color will soon change to a characteristic red. If a considerable quantity of nitrobenzole is present, the red color is changed to a more or less fine green color, which on the following day again becomes red. The quantity of nitrobenzole is determined by a violent and repeated agitation of the adulterated oil with four volumes of concentrated solution of sodium bisulphite. After some time rectified ether is added, which dissolves the nitrobenzole, and by evaporation permits its estimation. To prove the residue of the evaporation to be nitrobenzole, it is converted into aniline.

By another method the presence of nitrobenzole is ascertained as follows: Dissolve 1 part by weight of the suspected oil in 10 parts by weight of alcohol, compound the solution with 1.5 parts by weight of solid caustic potash, and evaporate the mixture to one-third its original volume. Pure bitter almond oil turns brown but remains fluid, while if adulterated with nitrobenzole a brown, resinous substance is formed, which floats in the liquid.

A new substitute for volatile oil of bitter almonds, observed in France during the past few years, is, according to Mr. Bryveau, neither cherry-laurel, peach-kernel, nor apricot-kernel oil. He gives the following points whereby the genuine oil and the substitute may be distinguished: Genuine oil of almonds has a specific gravity of 1.043; the substitute, 1.029 to 1.030. The substitute also has a less pleasant and a stronger odor. If the genuine oil is mixed with an equal volume of sulphuric acid, a handsome red color is produced, which becomes darker, but the oil remains thin, liquid, and clear. The substitute also forms a red mixture under these conditions, but this subsequently turns brown, turbid, and thick, and, after a few days, solidifies to a brown mass. The oils of cherry laurel, apricot and peach kernels behave much like oil of bitter almonds, though they become somewhat more rapidly dark red, particularly cherry laurel, and thicker; they, however, remain clear. The presence of 25 per cent of the substitute in any of these genuine oils causes the production of the brown color, as well as turbidity of the mixture.

Bitter almond oil is used in perfumery, in the preparation of liqueurs and confections, as well as in medicine. The essence of bitter almond oil used by perfumers is a solution of 0.35 to 0.70 oz. of the oil in 1 quart of alcohol.

*Bitter almond water* is the term applied to the watery distillate obtained as by-product in the preparation of bitter almond oil; it contains some benzaldehyde and prussic acid, and has a perceptible odor of almond oil.

*Almond oil* (*imitation of*), known in the trade as *nitrobenzole, oil of mirbane, essence of mirbane,* etc. This is benzol in which one atom of hydrogen is replaced by nitric peroxide or nitrogen tetroxide: $N_2O_4$ or $NO_2$. Its composition is therefore $C_6H_5.NO_2$. It closely resembles oil of bitter almonds in odor, but differs from it in other physical and chemical properties. It is prepared in the following manner: The upper end of a large glass worm is divided into two branches, gradually dilating so as to form two funnel-shaped tubes. Concentrated nitric acid is poured into one of the tubes and into the other, benzol. The two substances meet on the point of junction of the two tubes, the rate of flow being regulated by suitable means. Chemical reaction commences at once, the fluid becomes heated, and develops large quantities of suffocating vapors of hyponitric acid. For this reason it is best to carry on the operation in the open air. The product is repeatedly washed with water, which completes the operation. Nitrobenzole has a specific gravity of 1.209, boils at 415.5° F., and solidifies on cooling to 37.5° F. It is principally employed to scent soap. Its use for flavoring articles of food or drink should not be permitted, as, though opinions differ in regard to its poisonous properties, it is injurious to health.

*Cherry laurel oil, oleum laurocerasi* (*Kirschlorbeeröl,* G.;

*essence des feuilles de laurier-cerise*, F.) is obtained from the leaves of the cherry laurel, *Prunus Laurocerasus*, L., an evergreen shrub indigenous to Southern Europe. The leaves are bruised with water, allowed to stand in a moderately warm place for 24 hours, and then subjected to distillation.

Cherry laurel oil is yellow, resembles bitter almond oil in odor and taste, and has a specific gravity of 1.055 to 1.065 at 59° F. It does not occur ready-formed in the leaves, but the latter, according to Lehmann, contain 1.38 per cent. of amorphous amygdalin, the so-called laurocerasin of the formula $C_{40}H_{67}NO_{4} = C_{20}H_{27}NO_{11} + C_{20}H_{26}O_{13} + 6H_2O$, hence a mixture of amygdalin and amygdalic acid. It is evident that the amygdalin contained in laurocerasin will be decomposed by the action of emulsin like the glucoside of bitter almonds, but experience has shown the decomposition to progress somewhat more slowly. However, since it takes place by the simple maceration of the leaves with water, the leaves themselves must contain the ferment. The products of decomposition are, of course, benzaldehyde and prussic acid, of the latter about 2 per cent. of the oil. Hence cherry laurel oil and bitter almond oil would be identical. Tilden, however, claims to have discovered in the former benzyl alcohol, to which the slight difference in the odor may perhaps be due (Husemann).

*Wild cherry bark oil.* The bark of the wild cherry, *Prunus virginiana*, Mill., yields by distillation 0.2 per cent. of volatile oil, which consists chiefly of benzaldehyde, and contains much prussic acid. It has a specific gravity of 1.05 at 59° F.

### *Papilionaceæ.*

*Peru balsam oil* (*Perubalsamöl*, G.) Peru balsam is obtained from *Myroxylon Pereiræ*, Baill., a tree indigenous to San Salvador in Central America. At the beginning of the dry season, early in November or December, the bark for some distance up the trunk is beaten with the back of an axe or other blunt instrument until it has separated from the wood without breaking. Five or six days later the loosened bark is charred by means of torches, and in the course of another week the charred pieces fall off, when the bare wood is covered by rags which absorb the exuding balsam and which, when saturated, are gently boiled in water until most of the heavy balsam sinks to the bottom, the remainder being obtained by wringing the partly-exhausted rags in a bag made of stout rope. The balsam thus obtained is on the following day separated from the water and put into suitable vessels.

Peru balsam is viscous, of the consistence and appearance of molasses, of a dark brown-black color. It has an acid reaction, an agreeable, fragrant, slightly smoky odor, and a warm, bitterish taste, followed by a burning sensation in the fauces. It is heavier than water (specific gravity 1.15 to 1.16), and soluble in alcohol. The so-called *white Peru balsam* is derived, according to the older statements, from *Liquidamber styraciflua*, but according to more recent ones, by expressing the fruits of *Myroxylon Pereira.*

According to Frémy, Peru balsam contains cinnamic acid, resin and an oil—cinnamein or styracin. W. Richter found that an oily liquid separates when four parts of Peru balsam and 1 part of caustic potash dissolved in 1 part water are mixed by shaking, 2 or 3 parts of water added, and the whole allowed to stand. According to the quantity of water used, the oil floats on the surface or sinks to the bottom. It is of a yellow color, and tastes and smells somewhat of carrot oil. This *Peru balsam oil* has a specific gravity of 1.116 at 44.2° F., and is almost insoluble in water. It is constant in the air and not volatile with steam; heated by itself it does not pass over undecomposed. If the oil be vigorously shaken with 2 parts of 75 per cent. alcohol at 44.2° F., a portion of the oil dissolves, while another portion remains undissolved. The soluble oil Richter calls *myriospermin*, and the insoluble *myroxylin*; the former is almost colorless, while the latter is brown and becomes liquid at 54.5° F. K. Kraut arrived at somewhat different results. He prepared the oil by mixing the balsam with an equal weight of ether and shaking with the same weight of a 3 or 4 per cent. soda lye. After standing for some time, the upper layer was lifted off, washed, freed from ether by distillation, and dried at 248° F. in a current of hydrogen. Kraut thus obtained 60 per cent. of the balsam of oil. The alkaline solution contained resin, cinnamic acid and benzoic acid, while the Peru balsam oil contained: benzyl alcohol, $C_6H_5CH_2OH$, benzoic acid benzyl ester, $C_6H_5COOCC_6H_5CH_2$ (a colorless liquid of a slight odor, boiling at 577.4° to 579.2° F. and of specific gravity 1.114 at 65° F.), and cinnamic acid benzyl ester, $C_6H_5C_2H_2COOC_6H_5CH_2$. Hence, according to Kraut, it may be supposed that the small quantity of benzyl alcohol in Peru balsam oil is the result of decomposition.

Schimmel & Co. bring into commerce a volatile Peru balsam oil which is obtained by extracting the balsam, 100 parts of the latter yielding 0.4 part of oil, which consists chiefly of cinnamein (cinnamic acid benzyl ester). The oil is used in perfumery, in cases where an especially light color of the respective mixture is desired.

In conclusion it may be mentioned that Th. Peckolt has obtained from the leaves of Peru balsam tree a volatile oil consisting of two constituents and having a specific gravity of 0.874, and from the wood a similar oil having a sassafras odor and a specific gravity of 0.892 (Husemann).

*Tolu balsam oil.* (*Tolubalsamöl*, G.) *Tolu balsam* is obtained in a manner similar to Peru balsam from *Toluifera Balsamum*, L., a tree indigenous to Venezuela and New Granada. In a fresh state it is soft, but gradually becomes harder. It is of a brownish-yellow or reddish-brown color, a vanilla-like odor and a mild aromatic taste. It is readily soluble in alcohol, but almost insoluble in carbon disulphide, benzol and volatile oils; it is chiefly used as a fumigating agent. By distilling 4 parts of the balsam with 3 parts of water, the water of the distillate being constantly returned to the retort, 0.2 per cent. of volatile oil is obtained which contains cinnamein, benzoic acid and tolene. The latter may be obtained in a pure state by repeatedly rectifying the oil over caustic potash, and forms a thinly-fluid, colorless oil of a pungent, sharp pepper-like taste, and smells of elemi. According to Deville it boils at 338°F., and according to Kopp, at 309.2° to 320° F. On exposure to the air tolene is converted without coloration into a soft resin, while tolu balsam oil first deposits benzoic acid, and then becomes almost solid by resinification. According to Deville tolene has the formula $C_{11}H_{18}$, but according to Kopp, it is a terpene $C_{10}H_{16}$ (Husemann, Maier).

### *Cæsalpiniaceæ.*

*Copaiva oil, oleum copaivæ balsami,* (*Kopaivabalsamöl,* G., *essence de copahu,* L. This oil is obtained in the same manner as oil of turpentine from the balsam exuding from various species of *Copaifera.* The yield is 50 to 80 per cent. of the weight of the balsam.

Copaiva oil is water-clear, limpid, possesses an aromatic odor reminding one of the balsam, and a sharp, aromatic, somewhat bitterish and acrid taste. The oil has a specific gravity of 0.88 to 0.96, which, however, increases with age because the oil resinifies in the air. It boils between 473° and and 527° F. It is readily soluble in absolute alcohol and in ether, but only in 50 parts of alcohol of specific gravity 0.83. Ordinary commercial oil is levo-rotatory. However as shown by the considerable variations in boiling point, specific gravity, etc., the oils of different derivation are not entirely identical as regards their properties. The oil from ordinary copaiva balsam has, in a rectified state, a specific gravity of 0.878 to 0.881, boils at 473° F. and shows a rotatory power of —34° 18′, while oil from Para balsam has a specific gravity of 0.898, and a rotatory power of —28.553°. L. Poussett has examined an oil which was thickly-fluid, had a specific gravity of 0.91, boiled at 485.6°F., and was soluble with difficulty in absolute alcohol.

Copaiva oil consists of diterpene $C_{20}H_{32}$, sometimes mixed with diterpenehydrate $3C_{20}H_{32} + H_2O$. To the admixture of various quantities of the latter may perhaps be due the physical and partially chemical distinctions shown by copaiva oils of different derivations.

Copaiva oil is occasionally adulterated with gurjun balsam oil, the latter being even sold as a substitute for it. However, the price of copaiva oil is not high enough to make such sophistication particularly remunerative. On the other hand, copaiva oil is largely used for the adulteration of other volatile oils. According to H. R. Schramm, such sophistication is best detected by mixing a few drops of the oil to be tested with alcohol, saturating a wick with the mixture and igniting it. When the alcohol is burnt and the wick still glimmers, the odor of copaiva will be plainly perceptible.

Copaiva oil is used in medicine chiefly for gonorrhœa. Upon the human skin it acts very feebly as a irritant.

### IX. *Volatile Oils of the Families Ericaceæ, Oleaceæ, Convolvulaceæ, Labiatæ and Verbenaceæ.*

While the *Ericaceæ*, *Oleaceæ* and *Verbenaceæ* furnish but few plants from which volatile oil can be obtained, the *Labiatæ* comprises a large number of such plants which are of the utmost importance.

All the oils to be mentioned here, wintergreen oil alone excepted, occur ready-formed in the plants, and in most cases in special cells and glands of the leaves, and for this reason nearly all of them are obtained by distilling the entire plant with steam. The oils of this group are all fragrant and, by reason of a content of phenols, most of them are of medicinal, generally antiseptic value. Specifically heavier than water are wintergreen oil, and occasionally patchouli oil, all the others being specifically lighter than water.

As regards the composition of the oils to be discussed here, they are all oxygenated, with the exception perhaps of rosewood oil. *Hydrocarbons* are preponderatingly contained in oils of thyme, lavender and rosemary (pinene), further in oils of rosewood, horse-mint, savory, and occasionally peppermint. It may be mentioned as of special interest that the oils of marsh tea, patchouli and sage contain *sesquiterpenes.* The oils of wintergreen and andromeda consist chiefly of an *ester* (methyl salicylate); small quantities of esters occur in the oils of horsemint and American pennyroyal. Among the *oxygenated constituents* are to be mentioned: 1. Such of the formula $C_{10}H_{14}O$; in thyme oil (15 per cent.), horsemint oil (20 to 30 per cent.) and mother of thyme oil; in all three cases thymol; in spearmint oil and in crisped mint oil (25 to 50 per cent.), in these cases carvol; in Candia marjoram oil (50 to 80 per cent.), and in savory oil (30 per cent.) as carvacrol. 2. Such of the formula $C_{10}H_{16}O$: in European pennyroyal oil, and, as salviol, in sage oil. 3. $C_{10}H_{18}O$: lineol in the oils of spikenard, rosemary and sage. 4. $C_{10}H_{20}O$, menthol, in peppermint oil. 5. $C_{15}H_{26}O$, sesquiterpenehydrate, in the oils of patchouli and marjoram. The composition of some of the oils is not yet accurately known.

### *Ericaceæ.*

*Wintergreen oil, oleum gaultheriæ,* (*Wintergrünöl,* G.; *essence de gaultherie,* F.) is obtained by distillation from the wintergreen, *Gaultheria procumbens,* L., a plant common to North America, where it is otherwise known by the names boxberry, chequer-berry, partridge-berry, mountain-tea, tea-berry, etc. The apparatus used is, as a rule, very simple and crude, being easily taken down and removed to other parts when the supply of leaves is exhausted. This generally occurs in two or three years. The leaves are gathered principally by women and children. The price paid varies with the locality and abundance of the plant, seldom going below $1 or over $1.75 per 100 pounds. To gather 50 pounds of leaves is considered a good day's work.

The first point in locating, after finding a place where the plant grows in abundance, is to secure a stream of water with enough fall to allow it to be carried to the top of the cooling tub. Under a rough shed the apparatus is then set up, which usually consists of an ordinary copper whiskey-still with a capacity of 300 to 400 gallons, inclosed in brickwork, with the head only projecting. Others use a wooden box with a copper bottom and stayed with bolts. The head of the still is of copper, and connecting with this is a square or circular worm of the same material placed in a barrel. The leaves are placed in the still, covered with water, and heated by an open fire beneath. The steam carrying the volatile oil passes through the worm, is condensed, and the product is collected in the receiving vessel. The oil settling on the bottom, most of the water is dipped out and used again with the next lot of leaves, while the oil is collected by means of a separating funnel. The oil thus obtained is of a pale-red to deep-brown color, and by most manufacturers is thus sent into the market, packed in tin cans of various shapes and sizes. Some manufacturers obtain an entirely colorless product by filtering the oil through animal charcoal, while others have adopted the plan of macerating the charcoal with the oil for several days and then filtering off the oil, which is thus well decolorized. The wholesale dealers, who handle the oil in large quantities, generally decolorize by putting it in a bottle with crystals of citric acid and allowing the whole to stand, with occasional agitation.

According to J. E. de Vrij, the leaves of *Gaultheria punctata* and of *G. leucocarpa*, which grow especially on the summit of extinct volcanoes in Java, yield an oil identical with that of ordinary wintergreen. Wintergreen oil possesses a strong odor which, however, becomes agreeable by sufficient dilution; its taste is sweet, and at the same time warm aromatic. In water it is almost insoluble, but readily in alcohol and ether; however, it imparts to water its peculiar odor and taste. Its specific gravity is 1.18 at 59° F.; it boils, according to Proctor, at 411.8° F., according to Cahours, between 392° and 431.6° F., and, according to Power, between 424.4° and 429.8° F. The oil, according to Power, is slightly levo-rotatory, according to Schimmel & Co., optically inactive.

The first statements regarding the chemical nature of wintergreen oil were made by Cahours (1843). According to him the oil consists almost entirely of a body—methyl salicylate—boiling at 435.2° F. and having the formula $C_6H_4OHCOOCH_3 = C_8H_8O_3$. If wintergreen oil be moderately heated with potash lye of 45° to which pieces of solid potash have been added, a watery methyl alcohol distils off, while on adding a mineral acid to the residue in the retort, oxybenzoic or salicylic acid, which dissolves with difficulty in water is separated. Artificially-prepared methyl salicylate shows throughout the same properties as wintergreen oil. If the oil be saponified and acid immediately added without previously distilling off the methyl alcohol, the oil, accordiug to Cahours, separates again unchanged, salicylic acid separating out only the next day. Iodine is dissolved by the oil without change. It deserves attention that methyl salicylate is isomeric with anisic acid or methyl-paraoxybenzoic acid, $C_6H_4(OCH_3)COOH$, which was also recognized by Cahours. Finally, mandelic or phenylglycollic acid forms a third isomeric body of the formula $C_8H_8O_3$, but in this case equal $C_6H_5CH(OH)COOH$.

W. Procter has also studied the properties and reactions of wintergreen oil and shown that it gradually dissolves in ammonia solution, white crystals of salicylamide, $C_6H_4(OH)CONH_2$, being separated on evaporation. Procter further noticed the fact that the alkaline solution of wintergreen oil, on exposure to cold, gives with green vitriol a blue-gray precipitate; but by previously boiling the solution it is colored intensely red by the green vitriol (reaction of salicylic acid). Chlorine is absorbed with great avidity by the oil, vigorous heating and a yellow coloration taking place thereby; the oil finally solidifies, while chlorosalicylic acid, $C_6H_3ClOHCOOH$, is formed. Bromine yields a bromosalicylic acid under similar external phenomena, while iodine is dissolved without any external change in the oil. Cahours further found that methyl salicylate is converted by bromine into bromosalicylic methyl ester and dibromosalicylic methyl ester, both of them being crystallizing substances. In the same manner chlorine gives the methyl ester of chlorosalicylic acid, while fuming nitric acid yields, under violent reaction, first methyl nitro-salicylate, later on dinitro-salicylic acid methyl ester, nitro-salicylic and dinitro-salicylic acids. If wintergreen oil be distilled with barium or lime, anisol (methyl-phenyl ether), $C_6H_5OCH_3$ passes over, carbonate of barium or lime remaining behind.

Of greater importance is the fact that Cahours has also investigated the neutral substance, 10 per cent. of which occurs in addition to methyl salicylate in wintergreen oil. To obtain it wintergreen oil is mixed with potash lye and the mixture subjected to distillation. Methyl alcohol, water and the neutral substance called *gaultherilene*, which is insoluble in water, pass over. By washing with water the gaultherilene is obtained in a pure state; it is a colorless, very mobile oil of the formula, $C_{10}H_{16}$, boiling at 320° F., and having a strong, peculiar, pepper-like taste. By chlorine and bromine this terpene is converted into sticky products. According to F. B. Power and N. C. Werbke, oil of wintergreen contains only 0.31 per cent. gaultherilene, which, according to them, has a specific gravity of 0.94 and mixes with iodine without ignition. According to the more recent investigations (Chemical News, 1890) of F. B. Power, wintergreen oil consists of methyl salicylate with at the utmost 0.3 per cent. terpene, the latter forming a pale yellow, somewhat viscous fluid of specific gravity 0.94 and a pepper-like odor. Benzoic acid and the (ethyl) alcohol boiling between 152.6° and 163.4° F. are not contained in wintergreen oil.

According to what has been said, there can be no longer any doubt that wintergreen oil consists almost entirely of methyl salicylate with very small quantities (0.3 per cent.) of a terpene not yet thoroughly known.

Salicylate of methyl has recently been detected by E. Bourquelot (Comptes rendus, 119, 802) in the root of *Polygala vulgaris*, L., *P. depressa*, Wender., and *P. calcarea*, Schulz. as well as in the stalks of *Monotropa hypopitys*, L. Bourquelot considers it probable that, as is also the case with the bark of *Betula lenta*, the ester is not present in the above mentioned plants in the free state, but is formed by the action of a ferment upon a body analogous to Procter's gaultherin.

Salicylate of methyl appears to occur rather frequently in the vegetable kingdom; so far as known it has up to the present been found in:—

| | |
|---|---|
| *Gaultheria procumbens*, L. (flowers and leaves) | Cahours, 1843. |
| " *punctata*, Blume " " | de Vrij, 1871. |
| " *leucocarpa* " " | |
| " *Leschenaultii*, D.C. " " | Broughton, 1876. |
| *Betula lenta*, Willd. (bark) | Procter, 1844. |
| *Polygala Senega*, L. (root) | Langbeck, 1881. |
| " *alba* (root) | Reuter, 1889. |
| " *vulgaris* (root) | Bourquelot, 1894. |
| " *depressa*, Wender. (root) | |
| " *calcarea*, F. Schulz (root) | |
| *Monotropa hypopitys* (stalk) | |
| *Laurus Benzoin* (bark) | Schimmel & Co., 1885. |
| *Spiraea Ulmaria*, L. (flowers) | Schneegans and Gerock, 1892. |

But little genuine wintergreen oil is found in commerce, birch oil (from *Betula lenta*) being sold instead, the latter differing from it only in not containing the terpene.

The statement of P. Macewan that wintergreen oil is adulterated on a large scale with light camphor oil seems not very probable; moreover, the difference in the specific gravities of the two oils furnishes an excellent guide for the detection of such sophistication, camphor oil being lighter and wintergreen oil heavier than water. The behavior towards nitric acid may also be employed, camphor oil turning red with this reagent.

Oil of wintergreen is sometimes used as a carminative, but most frequently to conceal the taste of nauseous medicines. In the United States it is a popular remedy for almost any kind of disease. It possesses antiseptic properties, and a 2½ per cent. alcoholic solution of it has been recommended as an agreeable dressing for wounds. Wintergreen oil is also used for perfuming soap, and for flavoring purposes.

*Artificial wintergreen oil.* Since 1886, Schimmel & Co., of Leipsic prepare methyl salicylate and bring it into commerce under the name of artificial wintergreen oil. According to G.

M. Berringer, 10 parts by weight of salicylic acid are dissolved in 16 parts of absolute methyl alcohol and to the solution are added, inside of 24 hours, drop by drop, 18 parts of sulphuric acid, care being taken to avoid heating. The whole when thoroughly mixed is distilled from a retort in a current of steam.

According to Ch. Bullock, the artificial wintergreen oil brought into American commerce is a colorless liquid possessing the odor of the natural oil, a specific gravity of 1.176 and boiling at 398.3° F. On shaking a few drops of the artificial product with water the oil becomes turbid, while natural oil immediately separates clear.

*Andromeda oil.* According to J. Broughton, *Andromeda Lechenaultii*, a species of Ericaceæ, which grows in great abundance on the Neilgherry Mountains in India, yields a volatile oil consisting almost entirely of methyl salicylate and differing from wintergreen oil only in containing still less gaultherilene.

*Marsh tea oil, oleum ledi palustris,* (*Porschöl*, G.; *essence de lédon*, F.) is derived from the marsh-tea or wild rosemary, *Ledum palustre*, L., a species of Ericaceæ flourishing in marshy locations in the northern sections of North America, Asia and Europe. It is acrid-narcotic and is said to have been used in the manufacture of beer to render it more intoxicating. The volatile oil is obtained by distilling the flowering herb with water. It is of a slightly reddish yellow color, a sharp, pungent taste and a penetrating narcotic odor. It is scarcely soluble in water, but readily in alcohol and ether, and has an acid reaction. The yield amounts to 0.3 to 0.4 per cent. According to B. Ricca and A. Gorbow, marsh tea oil boils at 518° F., and consists almost exclusively of ledum-camphor. The latter, when heated in a closed glass tube with acetic anhydride to 302° F., yields a colorless liquid sesquiterpene, $C_{15}H_{24}$, of specific gravity 0.9349 at 32° F., and boiling at about 507.2° F.; it may, therefore, be considered as the hydrate of the sesquiterpene, $C_{15}H_{26}O = C_{15}H_{24}, H_2O$. The sesquiterpene itself forms the liquid portion of marsh-tea oil, so that the latter consists chiefly of ledum-camphor, $C_{15}H_{26}O$ (melting point 219.2° to 221° F.; sublimable in needles) and a small quantity of sesquiterpene, $C_{15}H_{24}$ (specific gravity 0.9349 at 32° F.; boiling point 507.2° F.)

### *Oleaceæ.*

*Lilac oil* (*Spanisch-Flieder, or Lilaköl*, G.). From the flowers of the lilac, *Syringa vulgaris*, L., is obtained by maceration or absorption the lilac pomade, the alcoholic extract of which forms lilac essence. The latter smells like tuberose, and is used as a substitute for tuberose essence, which is very scarce (*Hirzel, Piesse*). According to Favrot, a volatile oil may be obtained by extracting the flowers with ether, adding to the ethereal extract fresh flowers until it is saturated. By standing it separates into two layers, the upper one of which is lifted off and subjected to distillation. The distillate is allowed to stand in the air for the ether to evaporate, lilac oil, a thickish, amber-yellow mass, being obtained as residue. By filtering the latter a yellow liquid oil runs off, which smells of the flowers, and by standing separates more stearoptene, while a wax-like stearoptene remains behind upon the filter. It may be remarked here that the flowers of *Philadelphus coronarius*, L., also pass frequently under the name of "lilac" (French, "*seringa*"). However, they do not yield oil, but a pomade which serves as a substitute for orange-flower pomade (*Hirzel Piesse*).

### *Convolvulaceæ.*

*Rosewood oil or rhodium oil, oleum rhodii* (*Rosenholzöl*, G.; *essence de bois de rose ou de rhodes*, F.) is derived from *Convolvulus scoparius*, L., and *C. floridus*, L., two species of *Convolvulaceæ* indigenous to the Canaries. Both the root and the wood contain genuine rosewood oil. The oil is obtained by comminuting the wood or the roots, soaking in water and subjecting to distillation. The yield is 0.2 to 3 per cent.

Rosewood oil is somewhat thickly-fluid, pale yellow, becoming brown by age. It has an agreeable, rose-like odor and a bitter aromatic taste. It has a specific gravity of 0.9064 at 60° F., and boils at 480° F. It is miscible in all proportions with glacial acetic acid. It is levo-rotatory and, according to J. H. Gladstone, consists of 80 per cent. of a terpene, $C_{10}H_{16}$ boiling at 480.2° F., and smelling of sandal-wood and roses. Before the manufacture on a large scale of the oils of geranium and ginger-grass, rosewood oil was largely used as an adulterant of rose-oil; at the present time it is comparatively scarce in the market, and is chiefly employed for scenting so-called rose soaps. It is frequently adulterated with the oils of castor and copaiva. According to Schimmel & Co. commercial rosewood oil is generally only a mixture of various volatile oils, rose oil forming one of the constituents. Genuine rosewood oil is, according to their statement, a golden-yellow fluid of an agreeable rose like odor, and solidifies in acicular crystals at 53.6° F.

### *Labiatæ.*

*Thyme oil, oleum thymi,* (*Thymianöl,* G., *essence de thym,* F.) is distilled from the flowering herb of the thyme, *Thymus vulgaris,* L., a plant indigenous to Southern Europe, and elsewhere largely cultivated in gardens. The leaves possess a strong, aromatic odor. According to Flückiger, near Grasse, where the thyme grows wild, 88,000 lbs. of it are annually harvested and distilled in ordinary copper stills. The yield from the fresh herb is on an average 0.66 per cent. and from the dried herb 0.79 per cent. Fresh thyme oil is limpid, becoming somewhat more thick by age. In a crude state it is greenish yellow to red, when rectified, colorless, but becomes yellow to brown-red by standing. Its odor is strong and agreeable like that of the plant, and its taste, as a rule, camphor-like, cooling and somewhat pungent. Its specific gravity ranges from 0.87 to 0.904 at 59° F., the oil from fresh thyme possessing a higher specific gravity than the oil from the dried herb. Thyme oil is readily soluble in alcohol and ether, sparingly in water, and is levo-rotatory. Fresh rectified oil has a neutral reaction; old, red oil an acid reaction.

According to the various investigations of different chemists, thyme oil contains as chief constituents thymol, $C_{10}H_{14}O$ and thymene $C_{10}H_{16}$, and as a constituent of less importance cymol, $C_{10}H_{14}$. The thymene may be considered as levo-pinene. Regarding the proportional quantities of these three constituents little is known; according to Stohmann, the oil brought at present into commerce is frequently very poor in thymol, the reason for this being no doubt due to the fact the thymol is often extracted from the oil. According to Schimmel & Co., the content of thymol in oil distilled by them, which is said to be of better quality than French or Spanish oil, is about 15 per cent.

Thyme oil is occasionaly adulterated with oil of turpentine as well as with petroleum. The principal sophistication is, however, the withdrawal of thymol, whereby it loses its medicinal value, which is dependent upon the thymol. It is also used in perfumery, for instance, mixed with other odoriferous substances, for perfuming soaps, and in the preparation of mouth waters.

Thymol, $C_{10}H_{14}O = C_6H_3$ (1 : 3 : 4) $CH_3OHC_3H_7$, may be obtained by two different methods from thyme oil as well as from ajowan oil. 1. By cooling. 2. By shaking with soda lye, and decomposing the alkaline solution with hydrochloric acid. In Germany ajowan oil is obtained from Indian seed and chilled to crystallization. As a by-product thymene is obtained, which may be used for perfuming common soaps. Thymol melts at 111.2° to 122° F., and boils between 431.6° and 446°F. When pure it dissolves to a clear liquid in 5 parts by weight of 10 per cent. soda lye at 86° to 104°F. The solution is colorless or slightly reddish, but becomes darker on standing. If the thymol contains thymene, the latter, with this reaction, separates in the form of small drops of oil, the solution first becoming turbid. Thymol is chiefly used as an antiseptic.

*Horse-mint oil* (*Monardcn-* or *Pferdeminzöl*, G.) The horse-mint, *Monarda punctata*, L., is a perennial herb, indigenous to the United States, where it grows in dry sandy fields from New York westward to Illinois, and southward to near the

Gulf of Mexico. By distillation with steam it yields a volatile oil, which readily separates into a liquid and into a solid portion. The elaeoptene forms a yellow-red liquid smelling of thyme, and by rectification becomes pale yellow. The purified oil boils at 435.2° F., becoming thereby somewhat darker. On exposure to the air it readily resinifies and turns brown and thickly-fluid. The stearoptene forms large yellow crystals possessing an odor of thyme and yielding, after pressing and distilling with water, an oil which solidifies very slowly. In solidifying large regular rhombic laminæ are formed in addition to opaque, spherical, amorphous aggregates. If the crude stearoptene be distilled by itself, the distillate solidifies immediately. The purified stearoptene also possesses an odor of thyme, a pungent, acrid taste, melts at 118.4° F., and solidifies at 100.4° F. It boils at 428° F. It is readily soluble in ether and alcohol and, as regards its composition, corresponds to the formula $C_{10}H_{14}O$. According to Schimmel & Co., who have prepared the oil since 1884, it contains 20 to 30 per cent. thymol, being thus richer in this substance than thyme oil. According to H. Schrötter, horsemint oil contains 50 per cent. of a levo-rotatory terpene, $C_{10}H_{16}$, and up to 25 per cent. of dextro-rotatory thymol, $C_{10}H_{14}O$. The rest (25 per cent.) consists of various products of oxidation, a dextro-rotatory camphor $C_{10}H_{16}O$, as well as formic, acetic and butyric acids, the three acids being probably present in the form of esters, since the oil has a neutral reaction. Regarding the thymol, attention may especially be drawn to the fact that thymol from thyme oil is optically inactive. Further the thymol from fresh horsemint oil is said to be uncrystallizable, but crystallizes out with greater ease the older the oil becomes.

*Wild thyme oil* or *mother of thyme oil, oleum serpylli* (*Quendelöl*, G.; *essence de serpolet*, F.). Wild thyme or mother of thyme, *Thymus Serpyllum*, L., is indigenous to Europe and Northern Asia, and to some extent naturalized in North America. For the purpose of obtaining volatile oil the flowering herb is distilled with water or steam. The yield varies from 0.072 to 0.42 per cent., according to climate and locality, it being greatest from plants grown in a southern climate upon a soil rich in lime or marl.

Fresh mother of thyme oil is limpid and yellow to yellow-brownish; however, by age it becomes thickly-fluid and dark red. It has the somewhat lemon-like, agreeable odor of the plant, and a bitter, aromatic taste. Its specific gravity is 0.917 at 59° F. It is readily soluble in alcohol, while with water it forms a turbid fluid which slowly clarifies. Optical rotation, —10° 20′.

Wild thyme oil consists of cymol, $C_{10}H_{14}$, and thymol, $C_{10}H_{14}O$, besides a small quantity of cavacrol, $C_{10}H_{14}O$, and perhaps traces of a terpene, $C_{10}H_{16}$. Wild thyme oil absorbs iodine in considerable quantities, heating taking place thereby; at 185° F. the mixture ignites. Nitric acid, at the ordinary temperature, colors the oil dark brown and, on heating, resinifies it. Sulphuric acid thickens the oil, the mixture becoming dark brown-red to crimson. Wild thyme oil was formerly used as a carminative and as an addition to stimulating baths. At present it is seldom used except as a substitute for thyme oil in perfumery. In Paris the oil is prepared twice in the year, the first time in the months of May and June, and the second time in the fall.

*Peppermint oil, oleum menthæ piperitæ* (*Pfefferminzöl*, G.; *essence de menthe poivrée*, F.), from the fresh flowering peppermint, *Mentha piperita*, L. In the United States are cultivated and distilled annually, on an average, about 15,000 tons of peppermint plants, yielding about 100,000 pounds of oil. The average yield per acre of the crops of the first and second years is about 11 pounds; this would show an annual area under cultivation of about 9000 acres. The chief places of cultivation are Wayne county, New York, and St. Joseph and other counties, Michigan. The plant is also grown to a small extent in a few counties in Ohio, and in some parts of Upper Canada. At Mitcham, in Surrey, England, about 200 acres are devoted to this crop. Some peppermint is also distilled in Germany, in the region about Leipzig, and in France in the department of the Yonne, and in Southern India. The peppermint oil produced in China and Japan is characterized by the large proportion of stearoptene, menthol, or peppermint-camphor. In China the *Mentha javanica*, Bl., is the plant commonly cultivated, and its product consists almost entirely, it is said, of this camphoraceous substance. The cultivation of the peppermint plant and distillation of oil were introduced in Wayne county, New York, about 1830, and in St. Joseph county, Michigan, in 1835.

The plant should be grown in a warm, rich soil, which is not too dry, since it produces plants which are too small. On the other hand, plants grown in wet soil fail to produce oil in paying quantities. Gravel and clay are, therefore, unsuitable soils. The land should be well ploughed as late in the fall as possible, and either cross-ploughed or thoroughly pulverized in some other manner. In early spring the ground is marked out in furrows 2½ feet apart. In these furrows are placed the roots and runners which have multiplied from the setting of the preceding year. One acre of good roots usually furnishes sufficient to set from 5 to 10 acres of new ground. These roots and runners are from ⅛ to ¼ of an inch in diameter, and from 1 to 3 feet in length when in a healthy state. In setting they are carried in large sacks slung over the shoulders of the workmen, who place them in the rows so that there shall be one or two living roots or runners at every point in the row. While placing the roots with their hands, they at the same time cover them with their feet. A good experienced workman, in mellow ground, with good roots, can set about one acre per day. New plants appear above the ground about two weeks after setting, and are carefully hoed and cultivated until July and August, when, if the season is fair, the plants have thrown out such a quantity of runners as to render further cultivation very difficult, and indeed unnecessary. Great care must be had to keep the plants free from weeds. The weed which causes the most trouble is the *Erechites hieracifolia*, Raf., otherwise known as "broom-weed," "mare's-tail," "fire-weed," etc., a composite (like lettuce) yielding a volatile oil, which is bitter and pungent, and by its presence impairs the naturally fresh, penetrating, and delicious taste of pure oil of peppermint.

The harvest commences in August or as soon as the plant is in flower, by which time it will have attained a height of about two feet or upwards, and continues into September, warm or hot weather being essential at harvest time, that the plant may produce oil abundantly. The first crop is the best, the crop of the second year less desirable, and in the third year the ground may be again ploughed and the crop allowed to spring from the broken roots. The yield in the third year, when the ground is treated in this manner, is somewhat less than that of the first year. After this the land should be devoted for a time to some other crop.

For distillation the mint is cut down and allowed to wilt in the sun for five or six hours, and is then raked into "cocks," where it is allowed to remain for a short time before being distilled. Many growers, however, believing that a loss of oil is sustained by diffusion in the atmosphere if the plants are fairly dried, bring them to the still in a green state.

As distillation can be effected with much greater rapidity from dry plants, this question of thorough drying is one in which the owners of distilleries and the producers in general have long been greatly interested. For if the plants are worked in the green state it will require, ordinarily, about 90 minutes for distillation, with a yield of perhaps 5 to 7 pounds of oil to the charge; whereas, if thoroughly dried, distillation can be effected in about 35 minutes, with a yield of from 8 to 9 pounds, since dried plants will pack much more closely in the vats than green ones. Each owner of a distillery, on an average, distils the crops of ten other growers with his own, charging therefor 25 cents for each pound of oil obtained.

Mr. Albert M. Todd, of Nottawa, Michigan, the well-known grower of peppermint and distiller of peppermint and other oils, to whose able paper on " Oil of Peppermint," read before the meeting of the American Pharmaceutical Association, September. 1886, we are indebted for much information on this subject. has made careful experiments to test this question. In an article in the "American Druggist," September 1886, he says: "Upon a clear day in September, in the middle of the day, when no dew or moisture was present, two loads of peppermint plants were cut down side by side at the same time. Both loads were immediately raked up in the fresh state, containing all the natural juices of the plant, then drawn to the scales and weighed, One load was immediately distilled, the other load being spread upon the ground and dried for two days in the sun. At this time the plants had become freed from every particle of moisture, the leaves being so dry and brittle as to break off quite readily in handling. The second load, which had thus been dried in the sun and open air, was now spread out in a loft and exposed to a further drying and the action of the atmosphere for a little over six months.

"The first load of peppermint, which was distilled in the green state, weighed 2332 pounds and produced 6 pounds, 9 ounces of essential oil, being 1 pound of oil for each 355.35 pounds of plants, or 0.2814 per cent. After the second load had been dried and exposed to atmospheric action, as stated, for a little over six months, it was taken from the loft and distilled. The yield was 1 pound of essential oil for each 362.5 pounds of green plants, which slight loss—about 2 per cent. in amount of essential oil—is certainly to be accounted for fully by the portion of blossoms and leaves rattled off and lost in the numerous rehandlings. The charge of peppermint which was thus fully dried had shrunk 49.4 per cent. in its original weight.

"It will thus be seen that, although the plants are very aromatic, both before and after cutting there is no perceptible loss of the essential oil by diffusion in the atmosphere, the oil being so tightly held and 'hermetically sealed,' as it were, in its little prison cell, that a force greater than that existing in the atmosphere or the rays of the sun is necessary to free it. Indeed, leaves which fall off from the plants in dry seasons and remain upon the ground over winter, even though subjected to the action of rains and snows as well, are often found months afterwards to be so strong that one would hardly suppose that any of the strength had passed off. It is known, though, in practical experience, that when the plants are once thoroughly dried and subjected to rains, the water carries off a portion of the oil, acting in that respect as a distilling power of low degree."

In conclusion Mr. Todd says: "This experiment, combined with more extended experience in connection therewith, shows:—

"1. No loss of essential oil of peppermint through diffusion in the atmosphere is occasioned by thorough drying of the plants and prolonged exposure to atmospheric action prior to distillation.

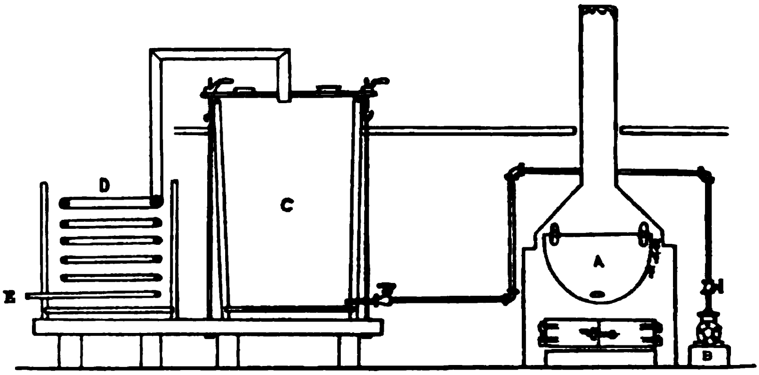
2. "Such exposure does not increase the crystallizing tendency of the essential oil.

3. "A heavy and insoluble resinoid is produced by oxidation, increasing the specific gravity of the oil and theoretically affecting the boiling point and solubility by raising the former and decreasing the latter.

4. "To obtain the best results as to the quality of oil produced and the facility of handling and distillation, the plants should be dried as thoroughly as possible without endangering the loss of leaves and blossoms; distillation should then take place as soon as convenient to prevent the formation of resin."

The apparatus and method of distilling in this country differ from those employed in Europe, where the fire is applied to the still. In this country the still consists of a wooden tub, or vat of heavy staves hooped with iron, and a size to correspond with the amount of steam furnished by a boiler. In every case two vats or still-tubs are arranged side by side, so that the process of distilling can proceed in one, while the other is emptied and refilled. Fig. 299 shows a still much in use in Wayne County, New York. *A* is in the steam boiler, *B* pump for supplying the boiler with water, and if need be for furnishing water to the worm tank, *C* still, *D* worm and worm tank, *E* end of worm from which condensed steam and oil are discharged. The worm is usually made of ordinary tinned plate iron, and from the commencement at the cover of the vat to its further extremity it gradually diminishes in size from 2½ or 3 inches to about 1 inch. The simplest mode of arranging the condenser is to lead it back and forth by means of angles through a running stream were possible, or, as represented in the illustration, coiled in a vat of planks, cold water being pumped into it by steam or by means of an hydraulic ram. The top of the still-vat passes through the floor above, from which the peppermint is introduced, and into which it is removed when exhausted of oil.

FIG. 299.



The wilted mint is packed into the vat by treading with the feet until the vat is full, when a cover, made steam-tight with rubber packing, is fastened down with screw-clamps. A steam-pipe connects the lower part of the vat with a steam boiler, and another pipe from the centre of the cover connects the vat with the condensing worm. The latter varies in size according to the capacity of the still, but becomes progressively smaller towards the outlet. The steam from the boiler being admitted to the vat at a pressure of 30 to 40 pounds, the oil of the mint is volatilized and mixed with the steam; it is condensed in the worm. The mixed oil and water are collected in the receiver, where the difference in their specific gravity causes them to separate. In many cases the receiver is a tin vessel, with a small pipe opening from its lower part and ascending nearly to the top, where it turns outward. The weight of the oil causes the water in the lower part to ascend in the discharge-tube until it overflows from the pipe. In the meantime the oil is dipped from the vessel whenever a few pounds have accumulated. The oil is packed in tin cans or glass demijohns holding about twelve pounds each. The glass demijohns are much the best, where the oil is to be kept any length of time. as its good qualities are more fully retained, and it is less liable to discoloration.

The exhausted mint is taken out with forks and dried for fodder. Cattle of all kinds prefer it to other fodder, but they will not touch the green herb. This fact is sometimes taken advantage of by turning sheep into a field of mint in order that they may clear it of grass.

In England the manner of distillation is as follows: The gathering of the peppermint crop commences in August and lasts for six weeks, the stills being kept at work day and night. These are of copper about 7 or 8 feet high and 5 feet in diameter, and holding about 5 cwts. of mint. A perforated bottom, provided with a large hook in the centre, for the withdrawal of the entire charge of herb by means of a windlass, rests in the still about 2 feet from the bottom, and enough water is poured in to cover the false bottom about 2 feet. The still is then filled with herb, which is trodden in by men, the lid, fitting into a water-joint, is fastened down by two transverse bars, and the distillation is then started by direct heat, requiring about 4½ hours. The yield varies according to the weather from 1 pound 12 ounces to 5 pounds per still, and being best when the summer has been dry.

The average price paid in the United States is from three to four dollars per pound for the crude oil, but the market has been known to jump one dollar per pound in a single day.

In Japan enormous quantities of peppermint oil are produced. but on account of its disagreeable bitter taste, which is probably due to a content of menthone, $C_{10}H_{18}O$, the oil is only suitable for perfuming soap and the production of cheap toilet articles. It may also be employed for the purpose of obtaining menthol. It is brought into commerce as "separated" or "unseparated" oil, or in crystals, *i. e.*, either only the liquid portion of the oil is sold or only the solid portion, or a mixture of both, the latter containing from 30 to 50 per cent. crystals of menthol.

Crude peppermint oil is of a greenish to yellow color; by shaking with animal charcoal or by rectification it is obtained colorless, but by standing it becomes again darker and even brownish. It has a penetrating peppermint odor and a pungent taste, imparting at the same time a sensation of coldness to the tongue and palate. Old oil, as well as Japanese oil, has a disagreeable, bitter taste. The specific gravity ranges from 0.84 to 0.961 at 60° F. Mr. Todd, in his paper already quoted, states that after many experiments he has come to the conclusion that the specific gravity of pure oils of peppermint, whether from English or American plants, should never be below 0.908, nor, when fresh and soluble, above 0.917, while the *true* boiling point should not be placed below 400° F.

The different peppermint oils, it is true, do not show any great difference in respect to specific gravity, but the variation is nevertheless sufficiently wide, often, to render this property indicative of the origin of the oil. The Japanese oil has the lowest specific gravity; it ranging, as a rule, between 0.895 and 0.905. The specific gravity of the English oil lies between 0.900 and 0.910, that of the American mostly between 0.910 and 0.920.

With 3 to 5 times its volume of alcohol of 70 per cent. at 68° F., English oil of peppermint gives a clear solution. If more alcohol be added, the solution generally remains clear, but often begins to show a slight cloudiness, which, however, should not reach the point at which oil-drops begin to separate. The solubility of Japanese oil in 70 per cent. alcohol is, as a rule, the same as with English oil, but often somewhat slighter.

In this instance it should be remembered that we are not dealing with a normal product, but with the residual oils of the menthol manufacture, which of course may vary in their properties according to the mode of their preparation. American oil is never quite soluble in alcohol of 70 per cent., but always leaves a certain proportion of insoluble parts, which separate from the rest as a layer upon the surface. Even in alcohol of 90 per cent. American oil of peppermint is not always soluble in all proportions. To attain perfect solubility at 68° F. ½ part by volume of alcohol is generally required. The United States Pharmacopœia justly demands that the oil shall give a clear solution with its own volume of alcohol of 90 per cent.

According to the United States Pharmacopœia peppermint oil must be soluble in carbon disulphide in any proportion, according to the Italian, it must form a cloudy liquid therewith, while the Belgian Pharmacopœia declares it to be only partially soluble in carbon disulphide. The following observations may explain these contradictory statements: If carbon disulphide be added drop by drop to any variety of peppermint oil, the first drops will cause a cloudiness which, at first, will disappear after shaking, but which reappears when more carbon disulphide is added. This cloudiness is caused by the water held in solution in the peppermint oil; it is not observed in an oil carefully dehydrated by means of anhydrous sodium sulphate. Hence the Belgian Pharmacopœia incorrectly regards the cloudiness as indicative of the imperfect solubility of the oil. The test is without practical object, as all possible adulterants are also soluble in carbon disulphide.

When 5 drops of American or English peppermint oil are mixed with 1 cubic centimeter of glacial acetic acid, a blue color begins to show itself after a few hours. This gradually acquires greater intensity, the maximum being reached in about 24 hours. At that time American oil shows a very deep blue color with magnificent copper-colored fluorescence. In the case of English oil the coloration is much weaker, often only reaching a pale blue color with faintly reddish fluorescence. Japanese oil alone does not give these reactions, the mixture remaining quite colorless.

Gentle heat or the immersion of the test-tube in boiling water considerably accelerates the reaction. When this is done, however, a much less pure blue color is obtained, the tint in that case being more violet.

Free access of air is absolutely necessary for the success of the reaction. If air is carefully excluded no coloration occurs, even with American oil, after several days. The reaction may, therefore, be considered a kind of oxidation process, a view which is supported by the fact that a similar color reaction is produced, but within a much shorter time, by applying the United States Pharmacopœia test of adding to 2 ccm. of the oil, 1 ccm. of glacial acetic acid and 1 drop of nitric acid. It should, however, be noted that this reaction also produces a slight violet tinge with Japanese oil.

According to the United States Pharmacopœia, oil of peppermint must separate menthol in a freezing mixture. If oil of peppermint is thoroughly cooled in a properly prepared freezing mixture of ice and salt, and a few menthol crystals added afterwards, the whole sample, in the case of American oil, generally solidifies, after a short lapse of time, to a solid mass. With English oil, however, no complete solidification occurs, as a rule, although considerable separation of menthol invariably takes place. Power and Kleber have shown that English oil of peppermint contains a greater proportion of alcoholic constituents than American oil. It may, therefore, be presumed that English oil contains certain liquid menthols, at present unknown, to which, perhaps, the excellence of its aroma is due. Japanese oil naturally separates little menthol in a freezing mixture, because it has previously been deprived of the bulk of that body.

As dementholized American peppermint oil is frequently met with in commerce, the freezing test is of importance in the case of this variety. The abstraction of a portion of the menthol would not be remunerative when applied to English oil, because that variety is much dearer than menthol itself.

The optical rotatory power of the various peppermint oils covers a rather wide range, hence only the grossest sophistication is able to alter the optical rotation of an oil to such an extent as to lead to detection by means of this test. Notwithstanding this fact we here subjoin, for the sake of completeness, the figures obtained by Schimmel & Co.* from the examination of a large number of specimens, from which slight differences between the special varieties themselves may be recognized. The optical rotation in a 100 millimeter tube and at about 68° F. is for

| American oil | English oil | Japanese oil |
|---|---|---|
| —25° to —33°. | —22° to —31°. | —26° to —37°. |

These figures show that the Japanese oil is optically the most active, English oil the least active.

Fresh peppermint oil has a neutral reaction, and old oil an acid reaction.

In view of the high importance attaching to the investigation of the composition of American peppermint oil, a synopsis of a treatise entitled: "On the Constituents of American Peppermint Oil and a Method for the Quantitative Determination of Menthol," by Dr. F. B. Power and Dr. Cl. Kleber, is quoted here from Messrs. Schimmel & Co.'s Report, October, 1894.

"Although oil of peppermint from different sources has repeatedly been the subject of chemical investigations, these have been restricted, for the most part, to the isolation and characterization of single constituents, without accomplishing the separation or identification of all the constituents of the oil. The chemists connected with the American laboratory of Messrs. Schimmel & Co. have therefore set themselves the task of filling this gap in our knowledge.

"The oils investigated were two samples prepared at the American works of Messrs. Schimmel & Co. from fresh and dried herb. The material was grown in Wayne Co., N. Y. It was taken from the first cutting of the season, and was quite free from foreign substances. The physical constants of the samples are given along with those of a large number of other peppermint oils in the table on p. 555. The presence of the subjoined bodies was proved:

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* Semi-annual Report, April, 1895.

| | | |
|---|---|---|
| 1. | Acetaldehyde | $CH_3—COH$. |
| 2. | Valeraldehyde | $(CH_3)_2CH—CH_2—COH$. |
| 3. | Acetic acid | $CH_3—COOH$. |
| 4. | Iso-valerianic acid | $(CH_3)_2CH—CH_2—COOH$. |
| 5. | Pinene (inactive?) | $C_{10}H_{16}$. |
| 6. | Phellandrene | $C_{10}H_{16}$. |
| 7. | Levo-limonene | $C_{10}H_{16}$. |
| 8. | Cineol | $C_{10}H_{18}O$. |
| 9. | Menthone | $C_{10}H_{18}O$. |
| 10. | Menthol | $C_{10}H_{19}—OH$. |
| 11. | Methylacetate | $C_{10}H_{19}—C_2H_3O_2$. |
| 12. | Methyl-iso-valerianate | $C_{10}H_{19}—C_8H_{11}O_2$. |
| 13. | Methyl ester of an acid | $C_8H_{12}O_2, C_{10}H_{19}—C_8H_{11}O_2$. |
| 14. | A lactone | $C_{10}H_{16}O_2$. |
| 15. | Cadimene | $C_{15}H_{24}$. |

"Although the determination of these bodies to some extent concluded the investigation, it seemed nevertheless of special interest to discover a method by the aid of which it would be possible to determine quantitatively in an accurate manner the chief constituent of oil of peppermint, viz., menthol (or the menthols *), and thereby to obtain a means of estimating the value of commercial peppermint oil; for it is sufficiently well known that much of the peppermint oil placed upon the market has been deprived of a portion of its menthol by exposure to cold.

### *Quantitative Determination of Menthol.*

"The most obvious method was that which had already done good service in the estimation of geraniol in geranium oils, namely, the conversion of the alcoholic constituents into the respective acetic acid esters, and by the decomposition of these by alcoholic alkali, and determination quantitatively of the amount of acetic acid which had entered into combination. In this way the menthol content of the oil could be directly ascertained.

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* Several isomeric menthols are believed to be present in oil of peppermint.

"In the first place it was determined that the esterification proceeded quantitatively, not only with pure menthol but also with mixtures of this with pinene, limonene and cineol; this was of course the first condition of the utility of the method.

"The estimation may be carried out with good results as follows:—

"About 20 grm. (accurately weighed) of peppermint oil, together with 30 ccm. of an alcoholic normal solution of sodium hydroxide (prepared by solution of 23 gm. metallic sodium in 950 ccm. of 95 per cent. alcohol and diluting with water to 1 litre) were either heated to boiling for an hour in a flask provided with a reflux condenser, or the mixture, contained in a strong, securely-stoppered glass bottle, was heated for an hour in a bath of boiling water, and subsequently the uncombined alkali titrated with normal sulphuric acid, with the use of phenolphtalein as an indicator.

"The saponified oil was then repeatedly well washed with water and finally boiled for an hour with an equal volume of acetic anhydride and 2 grm. anhydrous sodium acetate, in a flask provided with a suitable condensing tube, ground at one end so as accurately to fit the neck of the flask. The product, after cooling, was washed with water, then with a dilute solution of sodium hydrate, dried in contact with calcium chloride, and filtered. From 8 to 10 grm. of the resulting oil were then saponfied as above, with the use now of 50 ccm. of alcoholic normal solution of sodium hydrate, and the uncombined alkali determined by titration.

"As each cubic centimeter of normal alkali required for saponification corresponds to 0.156 grm. menthol, and as this yields 0.198 grm. menthyl-acetate, it is necessary, in order to calculate the found amount of menthol with reference to its percentage in the non-acetylized oil (free from ester), to subtract from the amount of oil used for saponification 0.042 grm. (the difference between 0.156 and 0.198 grm.), for each cubic centimeter of normal alkali consumed. If, for example, 8 grammes of acetylized oil had required for saponification *a* cubic centimeters of normal alkali, the total percentage P of menthol, free and in the state of ester, may be calculated by the following formula:

$$P=\frac{a\times 15.6}{S-(a\times 0.042)}.$$

"This does not indicate with absolute exactness the percentage of the menthol contained in the original oil, for it is assumed in this calculation that all the menthol which is present as ester is combined with acetic acid, whereas, as a matter of fact, it is partly in combination with iso-valerianic acid, etc. But the error so introduced is so small that it may be left out of account.

"As menthone may readily be converted into menthol by reduction, the above-described method was also employed for the determination of the amount of menthone in the oil distilled by the authors. The oil was saponified, and in a portion of the product, which had previously been deprived of alcohol, the percentage of menthol was determined. Another portion was diluted with twice its volume of alcohol, and treated at the boiling temperature of the liquid with metallic sodium. The results showed the following menthol-content: In the original saponified oil 54.5 per cent., in the portion once treated with sodium 67.0 per cent. and in that so treated twice 67.3 per cent. It could be assumed therefore that the reduction of menthol to menthone was complete. From these data the menthol-content of the oil came out at 12.3 per cent.

"Having a serviceable method of estimating menthol at hand, it appeared to be further of interest to ascertain whether the varying menthol-content was indicated by the fractional distillation, and if so, how. For this purpose a series of peppermint oils were fractionated in the following way:

"50 ccm. were placed in a fractionating flask provided with a high side-tube and distilled from a metal-bath in such a way that about one drop passed over per second. The temperature was read from a short thermometer the column of which was entirely immersed in vapor. The fractions that went over between each five degrees were collected and measured. The results of the distillation are given in the following table:

| Designation of the oil. | Specific Gravity at 59° F. | Optical rotation in a 100 mm. tube. | Menthol in the form of Esters. | Free Menthol. | Total Menthol. | From 50 ccm. of oil there distil in ccm. between the temperatures noted: | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | ? to 392° F. | 392° to 401° F. | 401° to 410° F. | 410° to 419° F. | 419° to 428° F. | 428° to 437° F. | 437° to 446° F. | 446° to 455° F. | Residue. |
| | | | % | % | % | | | | | | | | | |
| I. F. B., from dry herb | 0.9140 | —32° 0′ | 14.12 | 45.5 | 59.6 | 1.3 | 1.2 | 4.3 | 9.4 | 12.0 | 9.8 | 4.5 | 1.8 | 6.1 |
| Ia. F. B., from fresh herb. | 0.9130 | —30° 0′ | 11.25 | 43.2 | 54.5 | 0.6 | 1.2 | 5.7 | 8.5 | 11.6 | 9.2 | 4.2 | 2.2 | 6.7 |
| Ib. Wayne Co., N. Y., '92 | 0.9158 | —26°45′ | 9.32 | 40.8 | 50.1 | 1.4 | 1.8 | 6.3 | 10.0 | 12.9 | 7.3 | 2.4 | 1.8 | 6.3 |
| Ic. Wayne Co., N. Y., '93 | 0.9110 | —32°30′ | 9.04 | 46.1 | 55.1 | 1.1 | 1.4 | 5.0 | 10.7 | 13.9 | 7.6 | 2.9 | 1.6 | 5.4 |
| Id. Wayne Co., N. Y., Twice rectified (F. S. & Co.) | 0.9110 | —32°45′ | 8.61 | 51.0 | 59.6 | 1.5 | 3.7 | 8.8 | 14.7 | 12.2 | 4.2 | — | — | 5.2 |
| Ie. V. B. Co., Mich., '93 | 0.9067 | —29°20′ | 6.39 | 43.6 | 50.0 | 1.9 | 5.0 | 9.4 | 13.2 | 12.4 | 2.3 | — | — | 5.1 |
| If. Wayne Co., Mich., '93 | 0.9135 | —28°30′ | 7.73 | 50.3 | 58.0 | 1.7 | 1.6 | 5.5 | 9.6 | 12.2 | 8.9 | 3.2 | 3.1 | 5.8 |
| II. St. J. Co., Mich., '93 I | 0.9135 | — 9°45′ | 3.63 | 28.9 | 32.6 | 1.9 | 2.6 | 12.0 | 14.0 | 11.6 | 4.4 | — | — | 3.3 |
| IIa. St. J. Co., Mich., '93 II | 0.9083 | —19°30′ | 4.23 | 29.6 | 35.8 | 3.4 | 7.0 | 7.6 | 13.2 | 10.1 | 3.3 | 1.5 | — | 4.1 |
| IIb. "Rose Mitcham" | 0.9050 | —23°35′ | 4.37 | 44.2 | 48.6 | 4.4 | 9.6 | 13.4 | 17.1 | 3.2 | — | — | — | 2.5 |
| IIc. "Crystal White" Michigan | 0.9105 | —23°55′ | 6.74 | 44.2 | 50.9 | 1.7 | 5.8 | 9.9 | 15.4 | 9.4 | 3.4 | — | — | 4.1 |
| IId. "Redistilled oil" | 0.9105 | —23°30′ | 8.59 | 46.5 | 55.1 | 3.3 | 6.5 | 8.1 | 8.8 | 9.9 | 5.2 | — | — | 8.7 |
| III. Mississippi | 0.9250 | —13°40′ | 11.47 | 24.2 | 35.7 | 0.9 | 0.3 | 1.2 | 6.5 | 16.2 | 11.8 | 4.8 | 2.4 | 6.6 |
| IV. Japanese, normal. | 0.9100 | —34°45′ | 3.45 | 72.7 | 76.2 | 0.7 | 0.4 | 3.4 | 22.0 | 17.2 | 2.1 | 1.0 | — | 3.5 |
| V. " dementholized | 0.9030 | —31°20′ | 5.71 | 55.1 | 60.8 | 0.5 | 1.6 | 10.2 | 20.7 | 12.0 | 3.3 | — | — | 2.4 |
| VI. Mitcham. | 0.9070 | —27°55′ | 4.92 | 53.9 | 58.8 | 1.4 | 0.7 | 12.2 | 24.7 | 8.6 | — | — | — | 3.7 |
| VII. Saxon | 0.9100 | —26° 0′ | 6.38 | 61.2 | 67.6 | 1.7 | | 10.9 | 21.3 | 11.6 | 2.1 | — | — | 2.1 |

"The origin of the individual oils of the table is as follows:—

"I. F. B., from dry herb: Oil of Schimmel & Co.'s distillation from partially dry, Wayne Co., N. Y., herb.

"Ia. F. B., from fresh herb: Oil of Schimmel & Co.'s distillation from fresh, Wayne Co., N. Y., herb.

"Ib. Wayne Co., N. Y., '92: Oil from Wayne Co., N. Y., crop of 1892.

"Ic. Wayne Co., N. Y., '93: Oil from Wayne Co., N. Y., first cutting of new mint, 1893.

"Id. Wayne Co., N. Y. Twice rectified: Oil rectified by Messrs. Fritzsche Bros., and designated as F. S. & Co.

"Ie. V. B. Co., Mich., '93: Oil from Van Buren Co., Michigan, first cutting of new mint, 1893.

"If. Wayne Co., Mich., '93: Oil from Wayne Co., Michigan, first cutting of new mint, 1893.

"II. St. J. Co., Mich., '93, I: Oil from St. Joseph Co., Michigan, first cutting of new mint, 1893.

"IIa. St. J. Co., Mich., '93, II: Oil from St. Joseph Co., Michigan, second cutting of new mint, 1893.

"IIb. "Rose Mitcham:" Oil from a prominent distiller in Michigan. Stated to be obtained from Mitcham plants, improved by cultivation in America.

"IIc. "Crystal White," and

"IId. "Re-distilled Oil:" Both from the preceding Michigan distiller, and representing commercial oils.

"III. Mississippi: Oil from the State of Mississippi.

"IV. Japanese, normal: A normal Japanese oil.

"V. Japanese, dementholized: A Japanese oil, partially deprived of its menthol.

"VI. Mitcham: A rectified Mitcham oil.

"VII. Saxon: An oil distilled by Schimmel & Co., from plants cultivated by them at Miltitz.

"A consideration of the results given in the table leads to the following conclusions:—

"The specific gravity of peppermint oil varies between 0.905 and 0.916. A higher specific gravity has only been observed in the case of the Mississippi oil, and a slightly lower in the dementholized Japanese oil, which, of course, cannot be regarded as a normal peppermint oil.

"The optical rotation varies considerably, and does not seem to stand in any direct connection with the amount of menthol contained in the oil. It would appear, however, that the optical rotation of normal oils can be accepted as lying between —25° and —35°.

"The amount of menthol in the form of esters varies between 3.45 and 14.12 per cent. The normal limits of the amount of menthol in the form of esters which is contained in American peppermint oil may be accepted as from 6 to 10 per cent.

"The amount of free menthol is subject to considerable fluctuation, as it varies from 24.2 to 72.7 per cent. The content of normal American peppermint oil is always between 40 and 50 per cent. Oils containing a smaller amount, as those designated in the table II, IIa and III, may safely be considered to have been deprived of a portion of their menthol. Oils richer in menthol than the American are the Mitcham, the Saxon, and especially the Japanese.

"Although menthol is to be regarded as the most important constituent of peppermint oil, it should by no means be understood to represent the only factor which is to be considered in determining the value of the oil, as the odor and taste are very essentially influenced by the more or less favorable combination of the menthol with the other constituents of the oil. It is, for example, for this reason that the highly prized Mitcham oil, and above all that incomparably fine Saxon oil, possesses a much higher value than the Japanese oil, although the latter is much richer in menthol; they are, indeed, much more highly valued than pure menthol itself.

"The menthol-determination, however, as well as the fractional distillation, will reveal in many cases. whether an oil has been dementholized or adulterated."

Dementholized peppermint oil may also be readily recognized by its behavior in the cold. According to Schimmel & Co., the test is best applied as follows: Fill a test-tube nearly full with oil and place it in a mixture of equal parts of ice and common salt. In 10 to 15 minutes the oil should become wheyey, opaque and thick or jelly-like. Now throw in 4 or 5 small crystals of pure menthol, close the tube with a cork, shake vigorously and return it to the freezing mixture. In a short time the whole should be converted into a crystalline mass, otherwise the oil is dementholized.

Oil of peppermint found in commerce generally contains rectified spirit, and is frequently adulterated with oils of turpentine, spearmint, corn mint, etc.

According to Mr. Todd, oil of peppermint, when freshly distilled, or when two or three years of age, if kept in well-filled vessels, should dissolve readily in alcohol in all proportions, making a clear solution without need of filtration. When a few drops of the oil are placed upon white paper and held over a lamp or gas jet, it should volatilize quickly and perfectly without undergoing change or leaving any residue. When three drops of oil of peppermint are placed upon four grains of resublimed iodine there should be but a slight reaction, and what little vapor is produced should be almost invisible, becoming entirely so after having risen about twelve inches above the mixture, the color of the vapor assuming a bluish cast. The color of the mixture in this test should be carefully noted, which in the case of pure peppermint is of a brown or blackish-brown color, the iodine dissolving slowly and imperfectly. If oils of turpentine, erigeron, fire-weed, or other terebinthinate oils, are present, there will be quite a violent reaction, according to the quantity of the adulterant, with the evolution of considerable heat, and a red or reddish-yellow vapor will be produced, of a rank odor, partaking considerably of the nature of the adulterant, and the mixture will change to a bright violet. If the color of the mixture is most carefully noted, a very slight quantity of such adulterants can be detected. When to 25 drops of alcohol one drop of nitric acid of 1.2 specific gravity is added, and then one drop of pure oil of pepermint, there will be produced within about half an hour a lue or bluish-green color, which will remain permanent for a ong time. Oil of pennyroyal and of *Mentha arvensis*, or corn-nint, produce no coloration.

Another test for the detection of pennyroyal, which also indicates *Mentha arvensis*, when present in sufficient quantity, is as follows: Take one drachm each of chloral hydrate and pure sulphuric acid, adding twelve drops of alcohol. When this solution is mixed with a like quantity of pure oil of peppermint, a dark cherry color is quickly produced and maintained for a long time. Pennyroyal, or oil of peppermint heavily adulterated with it, gives no such color, being more of a yellowish cast and changing to olive green. With *Mentha arvensis* a yellowish-brown color is produced, which is maintained for 10 or 12 hours, and 36 hours later has a slight tendency to assume a cherry color or one intermediate between the cherry and the brown. It was noticed that, when the true oil of peppermint was mixed in equal proportions with that of the *Mentha arvensis*, a deficiency in the cherry color was plainly observable. To detect the presence of copaiba oil, boil a sample of the suspected oil with strong nitric acid. Pure oil of peppermint assumes a brown color, but remains limpid; if adulterated with copaiba oil, it becomes viscous and turpentine-like.

Oil of peppermint has many uses. It is employed for medicinal purposes in general, and is an important agent in cholera. It is also used in flavoring confectionery, perfumes, essences, and peppermint cordials, the latter being a favorite drink with Europeans. It is used as a household medicine all over the world, and for children's complaints it is unrivaled.

*Menthol*, $C_{10}H_{20}O$, has for a number of years been obtained from peppermint and brought into commerce by itself. According to A. M. Todd, the menthol is best obtained from the peppermint oil by first removing by fractional distillation the lower boiling portion, which consists chiefly of terpene. The residue is twice rectified until an oil very rich in menthol is obtained. The latter is cooled, whereby menthol crystallizes out.

The ordinary mode of preparation, however, is to simply cool the oil of peppermint and purify the separated crystals by centrifugence, pressing, etc., and under circumstances by re-crystallization from alcohol. The yield, according to Schimmel & Co., is about 50 per cent. for chemically pure material. Menthol forms colorless, lustrous, acicular or columnar crystals, possessing a strong odor of peppermint and a peculiar, aromatic taste. It melts at 109.4° F., boils at 413.6° F., and completely volatilizes in a water-bath. It should not produce coloration in a mixture of 1 cubic centimeter acetic acid with 3 drops of sulphuric acid and 1 drop of nitric acid. Menthol is largely used in medicine, and is frequently adulterated. Gorup-Besanez has shown that Japanese solid oil frequently contains as much as 13.66 per cent. of crystallized sulphate of magnesia, which crystallizes in a manner similar to menthol. A. Oppenheim also confirmed the existence of such sophistication of from 10 to 20 per cent. From England a cheap article contaminated with paraffine or camphor is brought into the market. Menthol pencils are sometimes adulterated with thymol, such sophistication, however, being possible only to a limited extent, since a mixture of 10 per cent. thymol and 90 per cent. menthol becomes salve-like, and one of 20 per cent. thymol and 80 per cent. menthol, jelly-like. According to Bernbeck, an admixture of thymol may be detected by the formation of a rose-red solution on heating 1 part of the suspected material with 4 parts sulphuric acid. If the solution be taken up with 10 times the volume of water, and digested with an excess of white lead, the filtrate acquires, by the addition of chloride of iron, a violet color. Leuken, on the other hand, recommends to dissolve the menthol in glacial acetic acid, add sulphuric acid, and then, drop by drop, nitric acid. The appearance of a blue coloration indicates the presence of thymol.

*Spearmint oil, oleum menthæ viridis* (*Grünminzöl*, G.; *essence de menthe verte*, F). This oil is obtained by distilling with steam the herb of the spearmint, *Mentha viridis*, L., which appears to be indigenous to England, but is cultivated and has been naturalized in the United States and most civilized countries. The oil resembles peppermint oil and has a specific gravity of 0.9142, but when freed as much as possible by distillation from stearoptene it has a specific gravity of 0.876 and then boils at 330.8° F. The oil freed from stearoptene possesses, according to Kane, the formula $C_{35}H_{50}O$. According to other statements, its specific gravity ranges from 0.91 to 0.93. According to J. H. Gladstone it consists of a turpentine-oil-like hydrocarbon in addition to a body isomeric with carvol, which has a specific gravity of 0.9515 and boils at 437° F. According to Trimble, spearmint oil contains a small quantity of a terpene identical with that of peppermint oil; further liquid carvol, $C_{10}H_{14}O$, which forms the chief constituent and does not solidify even at —9.4° F., but is precipitated by alcoholic ammonium sulphide; and finally a nearly colorless resin, which is partially formed during distillation.

Spearmint oil was formerly an article of commerce, but at present is not used by itself, though it is added to other kinds of peppermint oil, and sometimes is sold for crisped mint oil.

*Crisped or curled mint oil, oleum menthæ crispæ* (*Krauseminzöl*, G.; *essence de menthe frisée ou crépue*, F.) is obtained by distilling with steam the herb of the crisped or curled mint, *Mentha crispa*, L., and *Mentha crispata*, Schrad. The herb may be fresh or dried, but must not be too old. The yield from dried herb is 1 5 to 3 per cent., an average of 2.24 per cent. (Maier), or according to Schimmel & Co., 1 per cent.

Crisped mint oil is greenish-yellow to pale-yellow, becoming dark to red-yellow with age. Fresh oil is limpid, older oil more thickly-fluid. The oil possesses the odor of the plant and has a bitterish, aromatic taste, which is at first pungent and then cooling. Its specific gravity is 0.925 at 59° F. When exposed to a low temperature stearoptene is separated. Fresh oil has a neutral reaction; oil from old herb a slightly acid reaction. In alcohol of specific gravity 0.85, the oil dissolves in all proportions. Of the different commercial varieties American and German (Thuringia) oils are of about the same value, while Russian oil is of decidedly inferior quality. Corsican oil, which has recently been brought into commerce, has a greenish color and a strong aromatic odor.

Regarding the chemical nature of curled mint oil little is known, but it appears to be closely related to spearmint oil, the latter being, as a rule, sold for it. A. Bayer has obtained from the oils of curled mint and American spearmint a carvol, $C_{10}H_{14}O$. Iodine dissolves readily in the oil, heat being developed and violet vapors evolved. Nitric acid acts vigorously upon the oil and colors it first brown-red, after which resinification takes place. Sulphuric acid colors the oil dark brown-red.

Curled mint oil is much adulterated with alcohol, oils of turpentine, sassafras and eucalyptus, and especially with dementholized Japanese peppermint oil. According to H. Hager, oil of turpentine is, as a rule, not added to the oil, but French oil of turpentine is poured over the herb in the still, and is then difficult to detect by the odor, but always by the guaiac test. Curled mint oil is used for perfuming soap, but chiefly in the preparation of mouth waters and tooth tinctures.

*Pennyroyal oil* (*European*) *oleum menthæ pulegii* (*Poleyöl*, G., *essence de pouliot*, F.) The entire flowering herb of the European pennyroyal, *Mentha Pulegium*, L., is subjected to distillation with steam. The volatile oil obtained is yellowish, possesses a strong odor of the plant and a specific gravity of 0.927 to 0.939 for crude oil, and of 0.9255 for rectified oil. Williams gives the specific gravity as ranging from 0.925 to 0.939 at 60° F., and the boiling point at 418° to 420° F. Schimmel & Co., give the specific gravity of rectified Spanish pennyroyal oil as 0.945 at 59°F.; it boils between 356° and 446° F., more than 80 per cent. of it boiling between 428° and 446° F. In alcohol pennyroyal oil is readily soluble. Pulegon is one of the known constituents of the oil.

Pennyroyal oil absorbs, according to Williams, 1.46 to 4.62 per cent. of caustic potash, and 78.58 to 85.94 per cent. iodine. Of the commercial varieties the Spanish and Algerian oils are especially liked; American and French pennyroyal oils are dear. The oil is also produced in Russia, but from a different plant, viz. *Pulegium micranthum*, which grows in abundance on the steppes of Southern Russia, especially around Sarepta and Astrachan. *Russian pennyroyal oil* is limpid, and does not solidify even at 1.4° F. On exposure to the air it becomes more thickly-fluid and yellow-brownish. It possesses an odor between peppermint oil and curled mint oil. It passes almost completely over between 395.6° and 440.6°F., the distillate has a specific gravity of 0.932 at 62.6° F., and readily absorbs hydrogen chloride, and corresponds to the formula $C_{10}H_{16}O$. By oxidizing agents the body $C_{10}H_{16}O$ is converted into acetic acid, propionic acid, etc. Potash lye resinifies the oil. The use of pennyroyal oil has considerably decreased, dementholized Japanese peppermint oil having become a strong competitor with it.

*Pennyroyal oil* (American), *oleum hedeoma*, (*Hedeomaöl*, G.; *essence de pouliot americain*, F.). Pennyroyal, *Hedeoma pulegioides*, Pers., is an annual herb indigenous to North America, from Canada south through the United States. It grows in barren and sandy fields, on hills, and along the open borders of woods, always in dry places. The fresh herb is distilled with water or by means of steam, and yields a limpid, colorless or yellowish volatile oil of a peculiar pungent mint-like odor and taste. Its specific gravity is 0.93 at 59° F. The yield from dried leaves is 3 per cent., and from dried stems and leaves 1.3 per cent. The principal known constituent of the oil is pulegon.

American pennyroyal oil has a neutral reaction, is readily soluble in alcohol, and dissolves iodine with a brisk explosive reaction, yielding a viscous liquid. A mixture of the oil with chloral hydrate and sulphuric acid acquires a brownish-green color. The oil is used in medicine, it being an aromatic stimulant, carminative, and emmenagogue under fit conditions, and is added to embrocations to increase their anodyne and rubefacient action.

*Melissa or balm oil* (*Melissenöl*, G.; *essence de mélisse, de baume*, F.) Balm, *Melissa officinalis*, L., is a perennial herb indigenous to Western Asia and Southern Europe, and has become naturalized to some extent in the United States. By distilling the flowering herb with water a volatile oil is obtained, the yield from the fresh herb being 0.026 per cent. and from the dried herb 0.163 per cent. The oil is limpid, almost water-clear to slightly yellowish, has an agreeable lemon-like odor like the plant, and an aromatic, but not pungent taste. Its specific gravity ranges from 0.854 to 0.975. It has a slightly acid reaction and readily dissolves in alcohol as well as in 5 or 6 parts of spirit of wine of 0.856 specific gravity. On exposure to cold it separates stearoptene. Iodine dissolves in the oil under heating and the evolution of yellow, red and gray vapors, a soft, viscous residue being formed (Maier). The oil is often adulterated with lemon oil, though frequently an indifferent oil or one of a similar odor is distilled over balm herb and the product brought into commerce as melissa oil.

The oil from dried plants is said to be darker and of a less fine odor than that from fresh plants. Balm is chiefly cultivated in England. The oil is used in perfumery and rarely in medicine.

*Lavender oil, oleum lavandulæ* (*Lavendelol*, G.; *essence de lavande*, F). Lavender, *Lavandula officinalis*, Cha.. = *Lavandula vera*, D. C., is a native of Southern Europe, from Italy to Spain, and of northwestern Africa, growing upon sunny hillsides and mountains.

The plant is extensively cultivated in England, especially in Hertford and Kent, and in France, on the mountains which cover the southeastern corner, and in part of Piedmont, not farther west than the river Rhone and not beyond the 44th degree of northern latitude. The soil best adapted for lavender is a loamy one, with chalk subsoil, a warm situation sloping towards the south and perfectly free from trees. The soil must be in good condition previously to planting, and much labor is also required throughout the year to keep the ground perfectly free from weeds.

The method of cultivation and distillation in England is as follows: In making a plantation of lavender the ground is generally manured beforehand with 30 to 45 tons of stable manure per acre, but manure is not applied afterwards until a fresh planting takes place. The young plants are bedded out in November at a uniform distance of one yard apart; formerly they were planted at only half that distance, but it is found that a heavier yield is produced from plants set a yard apart than when planted closer together. When three years old the plants are at their best, and it is more profitable to uproot them and set a fresh plantation, the young plants yielding a much larger quantity of oil. When the plants are twelve months old, the whole ground is best forked between the rows of lavender, instead of ploughing, as some do, which prevents many of the small fibres from being cut into. In the autumn the plants are clipped with shears, which gives strength to them by keeping them nearer to the ground, and produces at harvest a larger crop of flowers.

The harvest time depends much on the state of the weather, but it usually commences about the first week in August. The flowers are cut with a sickle, bound up in sheaves weighing about 22 pounds each, and immediately carried to the distillery; there the stalks are cut off, leaving but little more than the flowers, by which the odor of the oil is much improved. Distillation is commenced about four or five o'clock in the morning, and the still, which has a capacity of from 200 to 1000 gallons, is filled four times a day, the work being continued until 10 P. M. The stills being filled tight to the top with the herb, liquor is allowed to flow in from the worm tub, which, though cold at the bottom, is boiling hot upon the surface. The furnace is then set to work, and the commencement of the distillation closely watched, that being the most critical stage of the process, resulting occasionally in the carrying over into the worm a portion of the solid contents. As soon as distillation commences the furnace is immediately damped. The water which comes over with the oil during the first hour being slightly impregnated with oil is returned to the still, but that which comes over afterwards is allowed to run away. The oil which comes over after the first hour and a half is either redistilled or sold as inferior quality. The refuse when removed from the still is thrown into heaps, and when decayed is returned to the lavender fields. The yield of oil from a ton of lavender averages from 15 to 16 pounds, but sometimes is not more than 10 pounds. The distillation is carried on from August to October. The oil improves by keeping up to three years, after which it begins to deteriorate unless mixed with spirit. Redistillation also improves its quality, but it is not profitable unless steam heat is employed, the loss being about one pound per gallon.

Oil of lavender, when freshly prepared, is a colorless liquid, which becomes yellow on standing. It has a hot, camphorous, slightly bitter taste and odor of lavender. It is levo-rotatory, has an acid reaction, and a specific gravity of 0.875 at 59° F. It begins to boil at 365° F., the temperature quickly rising to 374° F., and the greater portion distilling over between 383° to 419° F. The first portion consists of a mixture of acetic and formic acids, but contains no valeric acid. By repeated fractionation a levo-rotatory terpene, boiling at 323.6° F., is separated, capable of forming a crystalline hydrochloride. The essence also contains a mixture of camphor and borneol; this mixture forms an acetate, boiling at 446° F., which is decomposed by potash, yielding a terpene and potassium acetate. When it is distilled with phosphoric anhydride a hydrocarbon is obtained, consisting for the most part of terpene and containing also some cymene.

Lavender oil absorbs iodine with vigorous fulmination, yellow vapors being evolved and the oil being converted into a dark brown resinous mass. According to Williams, Mitcham oil absorbs 230 to 248.6 per cent. of iodine; Hitchin oil, 233 per cent.; and French oil, 199 to 237 per cent.

G. Massol (Journal de Pharmacie et de Chimie, 1895, 49) has communicated certain data concerning the specific gravity and the rotatory power of lavender oil and spike oil, and compares his results with those obtained by other French investigators. The figures of the different authors vary widely, as is seen from the following synopsis:

| | | Massol | Buignet | Bruylants |
|---|---|---|---|---|
| Lavender oil. | sp. gr. at 59° F. | 0.930 | 0.886 | 0.876 |
| | opt. rot. $\alpha_D$ | —3°.70 | —21°.20 | —2°.29 |
| Spike oil .... | sp. gr. at 59° F. | 0.935 | 0.866 | 0.908 |
| | opt. rot. $\alpha_D$ | +9°.66 | +3°.30 | —0°.34 |

According to the investigations of Schimmel & Co., which have been made upon hundreds of samples, and which were completed, in the case of lavender oil, by the determination of the ester content, and in that of spike oil by the determination of the alcohol content, as well as by solubility-tests, a good lavender oil has a sp. gr. of 0.885 to 0.895. Its opt. rot. varies from —3° to —8° in a 100 mm. tube. The sp. gr. of a good spike oil is 0.905 to 0.920, opt. rot. +1° to +9° in a 100 mm. tube.*

Oil of lavender is frequently adulterated, chiefly with alcohol, but occasionally with oil of bergamot. The pure oil fulminates quickly and violently with iodine, and sulphuric acid turns it reddish-brown, the reaction being accompanied by strong thickening. An admixture of alcohol is readily detected by treatment with a small quantity of tannin. If the latter is not altered, the oil contains no alcohol; if, however, it becomes viscous and sticky, it is adulterated. Adulteration with oil of turpentine is detected by treatment with strong alcohol. For the complete solution of 1 part of oil of lavender 5 parts of 90 per cent. alcohol are required; if, however, the oil contains turpentine, the fluid is turbid.

Lavender oil by itself is only used for the finest perfumes and soaps. It is chefly employed in preparing essences and lavender water.

*Spike lavender oil, oleum spicæ*, (*Spiklavendelöl*, G.; *essence*

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* Semi-annual Report of Schimmel & Co., April, 1895.

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*d'aspic*, F.) The spike lavender, *Lavendula Spica*, D. C., grows everywhere where genuine lavender occurs, and serves also for obtaining volatile oil. Spike lavender has a strong, but less agreeable odor than *Lavendula vera*, and for the purpose of gaining oil is chiefly gathered in Southern France. The yield from the fresh plant is 0.45 to 1 per cent. The oil is distilled in September.

Spike lavender oil is limpid, becoming thicker by exposure to air and by age; when fresh it is almost colorless, later on brownish, possesses a penetrating aromatic odor resembling somewhat that of wild thyme, and has a neutral reaction. Its specific gravity ranges from 0.905 to 0.920 at 59° F. It boils at 366.8° F.; according to Williams, between 330° and 339.8° F. Optical rotation + 1° to + 9°. It is readily soluble in alcohol; on cooling it separates, according to Kane, large masses of stearoptene.

The chemical composition of spike lavender oil is not yet thoroughly known; however, it is certain that the oil contains varying quantities (35 to 70 per cent.) of terpene, probably dextropinene, further cineol, probably mixed with a camphor (55 to 20 per cent. of the mixture), and finally a resin (10 per cent.)

Towards iodine spike lavender oil behaves in a manner closely resembling that of lavender oil, but the reaction takes place with less violence. Williams found that the oil absorbs from 207 to 288 per cent. iodine. Of caustic potash it takes up 0.74 to 1.99 per cent. Nitric acid only colors the oil slightly yellowish, but, on heating, resinifies it. Sulphuric acid colors it yellow-brown.

Spike lavender oil is adulterated with oil of turpentine, as well as with alcohol and fixed oils; it is, however, more frequently used for the adulteration of other oils, especially of genuine lavender oil.

*Rosemary oil, oleum rorismarini, sive anthos* (*Rosmarinöl*, G.; *essence de rosmarin*, F.) Rosemary, *Rosmarinus officinalis*, L., is a native of the basin of the Mediterranean and is extensively grown in Italy, Southern France, Greece, Egypt and Algiers, and to some extent in England. The best quality of rosemary oil comes from Southern France, the so-called Italian oil, which is probably exclusively produced in the Dalmatian islands, forming the inferior quality of commerce.

The rosemary is found in special abundance in the Dalmatian island, Lesina. According to C. O. Cech, every third year, during the month of May, the biennial shoots of the plant are cut off with knives, dried for about eight days in the sun, and deprived of their leaves. The latter are then moistened with water and transferred to copper stills, which are heated over an open fire. The volatile oil passing over with the steam is condensed in the usual manner and received in flasks. As soon as the oil is separated from the water it is poured into tins and shipped to market.

It appears that in France only the flowering points of rosemary branches are used for distillation, which, together with the better apparatus, may account for the superiority of the French article. The yield is 1.2 to 2 per cent., the content of oil in the plant appearing to decrease in a northern climate.

Rosemary oil in a fresh state is colorless and limpid, but becomes darker and thicker by age. It possesses a penetrating odor of rosemary and an aromatic, pungent, camphor-like taste. The specific gravity of the commercial oil ranges from 0.88 to 0.915, and that of the rectified product from 0.885 to 0.887. According to Williams, the specific gravity of the oil is 0.894 to 0.912 at 60° F., while Schimmel & Co.* give the following specific gravities: Oil from fresh flowering herb 0.90 to 0.91; from dried Italian herb 0.912; from dried French herb 0.914; from dried French flowers 0.920. Bruylants gives 302° to 500° F. as the boiling point of an oil of specific gravity 0.885 at 56.3° F.; and Williams, 335° F., as that of an oil of specific gravity 0.894 at 60° F. In distilling the oil 90 per cent. by volume pass over, according to H. Eckenroth, up to 347° F., while the official directions for testing rosemary oil used as an addition to alcohol require that at the utmost 5 per cent. by volume shall distil off up to 320° F., and at least 90 per cent. by volume up to 392° F. According to Eckenroth, a clear solution should result by mixing at 68° F. 10 cubic centimeters of rosemary oil with 120 ccm. of 73.5 per cent. alcohol. In a fresh state rosemary oil has a neutral reaction, and when old an acid reaction. It turns the ray of polarized light to the left.

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* Report, October, 1893.

Rosemary oil consits of a levo-terpene, $C_{10}H_{16}$ (80 per cent.), included in which is levo-pinene; further of dextro-borneol and levo-borneol (0.5 to 0.65 per cent.), dextro-camphor and levo-camphor (9.5 to 12.35 per cent.), and finally cineol. The levo-terpene may probably be entirely levo-pinene, but sufficient proofs for this assumption are wanting.

Rosemary oil absorbs 142 to 162 per cent. of iodine with perceptible heating and evolution of yellow red vapors, a viscous, yellow-brown residue being formed. Hydrogen chloride is absorbed in large masses by the oil. Sulphuric acid colors the oil brown-red and thickens it. Nitric acid causes the formation of a brown-red balsam, whereby heating takes place and yellow vapors are evolved. The absorption of caustic potash by the oil amounts to from 0.78 to 0.88 per cent. Chromic acid converts the oil to a resin-like mass of an acid character.

Rosemary oil is frequently adulterated with oils of turpentine, spike lavender, petroleum, alcohol, etc. Sophistication with oil of turpentine is generally effected by distilling oil of turpentine over rosemary herb, and selling the product as rosemary oil. Such adulteration may be readily detected by the behavior of the oil towards iodine and nitro-prusside of copper, and its slight solubility in 90 per cent. alcohol. Petroleum is used as an adulterant, especially in England; according to R. A. Cripps it may be recognized by the odor on evaporating the oil in a water bath. Further, normal rosemary oil dissolves in 5 parts alcohol of specific gravity 0.838, while oil adulterated with petroleum requires 20 to 30 parts alcohol, and besides the solution, shows a yellow color and fluorescence. With alcohol of 0.748 specific gravity, rosemary oil containing petroleum becomes turbid. An addition of alcohol is indicated by the increased solubility of the oil in alcohol, and, in addition, such oil is colored red by magenta-red.

Rosemary oil is one of the principal ingredients of eau de cologne and the once famous Hungary water, and is also used in the manufacture of liqueurs, aromatic waters and for scenting soaps.

*Basilicum oil, or sweet basil oil (Basilikumöl,* G.). By distillation with water sweet basil, *Ocynum Basilicum*, L., yields about 1 ½ per cent. of volatile oil; according to Schimmel & Co., the fresh German herb yields only 0.02 to 0.04 per cent. The oil possesses an aromatic smell and on standing separates prismatic crystals of a stearoptene. The latter has been examined by Dumas and Peligot, who found it to be of the composition, $C_{10}H_{22}O_3 = C_{10}H_{16}+3H_2O$. This basilicum camphor forms, according to Bonastre, four-sided columns or colorless, transparent plates, according to whether it has been recrystallized from alcohol or boiling water. It is almost colorless and tasteless, and has a neutral reaction. It readily dissolves in hot water or hot alcohol, in ammonia and acetic acid, and in 6 parts of ether. Sulphuric acid colors the crystals red. The specific gravity of the oil from fresh German herb has, according to Schimmel & Co., a specific gravity of 0.918 to 0.928, and the oil from Reunion herb, one of 0.946 to 0.967 at 59° F. The optical rotation of the latter is $+16°$.

*Patchouly oil, oleum patchouli, (Patschuliöl,* G.; *essence de patchouly,* F.), is cbtained from the leaves of *Pogostemon Patchouly,* Pell., which posseses a musk-like odor. In Singapore, where the plant is largely cultivated, the oil is prepared by submitting the leaves to distillation. The leaves are first dried in the shade on bamboo racks. During this process they are frequently turned over, and, when so far dried as to have just sufficient moisture to permit of a slight fermentation, they are piled in heaps and allowed to heat gently; after this they are spread out and dried, but not to absolute dryness, and are immediately distilled. The distillation is effected by passing steam, generated in a boiler apart, through the leaves in the stills. The pressure of steam is not allowed to rise above 30 pounds. The yield under these conditions is about one-quarter ounce per pound of leaves; by high-pressure steam, the yield would be greater, but it is rank in quality. Baled leaves are now sent to Europe and distilled there, but the oil obtained from them is not so good as the Singapore oil, owing to the fact that the leaves exported are mostly from the wild plant.

In commerce, several kinds of oil are distinguished, viz. Penang oil and French oil, and in Penang itself, the green and brown oils, the former being in greater demand.

Patchouli oil is yellowish green to olive brown. The thickly-fluid green oil is said to be obtained from dried leaves, and is claimed to possess a different odor from the brownish Singapore oil. The odor of the oil is penetrating, somewhat musty and, in an undiluted state, disagreeable, it becoming agreeable only with sufficient dilution, or when mixed with other odoriferous substances. The specific gravity of the oil is 0.97 to 0.99 at 59° F. Between 539.6° and 561.2° F., the oil passes almost completely over. It dissolves completely in equal volumes of 90 per cent. alcohol. By long standing it separates patchouly-camphor, the latter possessing a specific gravity of 1.05.

Patchouli oil consists of sesquiterpene, $C_{15}H_{24}$ (boiling point, 525.2° to 527° F.; solid dihydrochloride fusing at 242.6° to 244.4° F.), and patchouly camphor, $C_{15}H_{26}O$, besides a certain amount of coerulein. The proportional quantities of terpene and camphor vary; however, the importance of the oil as an odoriferous substance appears to be dependent on the content of sesquiterpene. The above-mentioned formula for the camphor is the more likely, since it represents the hydrate of the sesquiterpene ($C_{15}H_{26}O = C_{15}H_{24},H_2O$).

By boiling with potash lye patchouly oil becomes pale brown; concentrated sulphuric acid colors it, when much diluted, dark red-brown; nitric acid also colors it dark red-brown, and, on heating, converts it into a red-brown resin which dissolves in alcohol, the solution acquiring a red color. Hydrochloric acid colors the oil dark crimson; by the addition of a little alcohol the mixture becomes dark blue-violet, and isolated drops of oil separate on the surface.

So far as known patchouli oil is adulterated chiefly with cedar-wood oil. It is used only in perfumery, though the Arabs, who are very fond of the perfume of patchouli, ascribe remedial powers to the herb. The powerful odor of patchouli oil is best shown by the fact that when mixed in the proportion of volume for volume, it completely covers the smell of all other bodies. Only when added in very small quantities to other odoriferous substances new combinations of a peculiarly agreeable odor are formed. Chinese ink is perfumed with a mixture of camphor and patchouli oil. Powdered patchouli herb serves for perfuming clothes and clothing, it protecting the latter, it is said, from moths.

*Sage oil, oleum salviæ* (*Salbeiöl*, G.; *essence de sauge*, F.) Sage, *Salvia officinalis*, L. is a suffructicose perennial, indigenous to Southern Europe and extensively cultivated in England, France and Germany, and in gardens in the United States. The leaves have a balsamic, aromatic odor and a similar, but somewhat harsh and astringent taste. Besides tannin and a bitter principle they contain a volatile oil which is obtained by distilling the fresh or dried herb with water or by means of steam. The yield is from 1 to 1.4 per cent.

Freshly-prepared sage oil is pale yellow, yellowish-brown to greenish-yellow, sometimes almost colorless. By standing it becomes darker. It is limpid and possesses a penetrating sage-like odor, which becomes agreeable on dilution, and a somewhat sharp, pungent taste. Its specific gravity ranges from 0.86 to 0.92. It boils at from 266° to 320° F. It is soluble in all proportions in alcohol. It is dextro-rotatory. By standing it separates stearoptene. Fresh oil has a neutral reaction; old oil an acid reaction.

Sage oil contains pinene, $C_{10}H_{16}$ (partly levo-pinene, partly dextro-pinene, partly an inactive mixture; boiling points given at 305.6° to 312.8°, 314.6° to 315.5°, and 316.4° to 334.4° F.); further nominally a second terpene, $C_{10}H_{16}$ (boiling point 323.6° to 334.4° F.; levo-rotatory), small quantities of cymol, $C_{10}H_{14}$, and cineol, $C_{10}H_{18}O$ (fraction between 345.2° and 352.4° F.); as chief constituent, salviol, $C_{10}H_{16}O$ (boiling points given at 386.6° to 203° and 393.8° to 399.2° F.; dextro-rotatory); further sage-camphor, $C_{10}H_{16}O$ (melting point 345.2° to 366.8° F.; boiling point 401° to 410° F.; inactive); finally sesquiterpene, $C_{15}H_{24}$ (cedrene; boiling point 500° to 519.8° F.) According to this, the oil has an extraordinarily complicated composition, but the statements in regard to it are somewhat contradictory. Muir, for instance, asserts that sage oil contains but little salviol, but much sesquiterpene, at least English oil, while Wallach names salviol the principal constituent of the oil. The statements regarding the pinene occurring in sage oil are also contradictory, Muir finding it now levo-rotatory and again dextro-rotatory, while Wallach calls the respective fraction inactive.

Iodine dissolves quietly in sage oil, an oil of 0.961 specific gravity dissolving, according to Williams, only 49.7 per cent., and an oil of 0.918, specific gravity, 117.14 per cent. Of caustic potash the oils absorbed 7.09 per cent. and 1.58 per cent. respectively. Williams explains this by stating that fresh sage oil contains but little salviol and camphor, but is rich in cedrene, while in old oil the reverse is the case.

The only known adulterations of sage oil are an admixture of lavender oil whereby it is said to become greenish-yellow and turbid, as well as specifically lighter, and of oil turpentine, which may be detected and isolated by distillation. The oil is not much used, though it may be employed for perfuming soap.

*Hyssop oil, oleum hyssopi* (*Ysopöl*, G.; *essence d'hysope*, F.) Hyssop, *Hyssopus officinalis*, L., is indigenous to Southern Europe, and has a camphor-like, aromatic odor. By distilling the herb a volatile oil is obtained. The yield from the dried herb is 0.7 to 4 per cent. The oil is colorless but on exposure to the air soon turns yellow, and resinifies. Its specific gravity ranges from 0.889 to 0.986, and it boils between 287.9 and 325° F. It is soluble in an equal weight of alcohol of 0.85 specific gravity. Hyssop oil is occasionally used in France for cheap perfumes, but chiefly in the manufacture of liqueurs.

*Origanum oil* (*common*) *or wild marjoram oil* (*Gewöhnliches Dostenöl*, G.) Origanum or wild marjoram (*Origanum vulgare*, L.) is a perennial herb growing in dry soil throughout a large portion of Asia, Europe and North Africa, and has also been introduced into the United States. The herb has an agreeable aromatic odor and an aromatic bitterish taste. It contains tannin and a volatile oil, the latter being obtained by subjecting the flowering herb to distillation with water. The yield is 0.5 to 3 per cent.; the richness in oil of the plant appearing to increase towards the north. The oil is straw-yellow, becoming brown-yellow with age, limpid, of a mild aromatic odor and bitterish, sharp aromatic taste. Its specific gravity ranges from 0.8673 to 0.909. The rectified oil boils almost constant at 321.8° F. Origanum oil has a neutral reaction and dissolves in every proportion in absolute alcohol, but only in 12 to 16 parts of alcohol of specific gravity 0.85.

By iodine origanum oil is converted into a thickish, dark brown body, a very vigorous reaction taking place thereby. Nitric acid colors the oil brown red and, on heating, resinfies it. Sulphuric acid thickens the oil somewhat and imparts to it a dark blood red color.

Origanum oil is used in pharmacy.

*Origanum oil (Cretic) (Kretisches Dosten- or Spanisches Hopfenöl*, G.). Under this name is brought into commerce an oil obtained from a series of varieties of Origanum *(Origanum hirtum*, Koch.; *O. smyrnæum*, Benth.; *O. creticum)* regarding the properties and composition of which the statements are partially contradictory. The oil when fresh is generally yellow, when old red-brown. It is limpid, of a penetrating aromatic odor, and a sharp, pungent taste. It has a neutral reaction.

According to E. Jahns, the principal constituent of the oil from *Origanum hirtum* is carvacrol, $C_6H_3(OH)C_3H_7CH_3$, identical with that from caraway oil. It may be readily separated by shaking the oil with soda lye, acidulating the alkaline liquid with sulphuric acid, and purifying the separated carvacrol by rectification. In addition, Cretic origanum oil appears to contain another carvacrol, further terpenes, and, perhaps, a small quantity of cymol. An oil prepared by Gehe & Co. was reddish yellow, not very limpid, of thyme-like odor and specific gravity, 0.951 at 59° F. It turned the polarized ray of light slightly to the left, and contained 50 to 60 per cent. carvacrol. A presumably Greek oil was red brown, had a slightly acid reaction, specific gravity 0.966 at 59° F., and contained 70 per cent. carvacrol. An oil from Asia Minor had a pale yellow color, was slightly turbid and thickly-fluid. Its specific gravity was 0.95, it was levo rotatory and contained 50 to 60 per cent. carvacrol. An oil from Chios, distilled in Trieste, was light red brown, somewhat thickly-fluid, slightly levo-rotatory, of specific gravity 0.978, and contained nearly 80 per cent. carvacrol.

The brown oils are likely derived from *Origanum smyrnæum*, L. The French oil comes into commerce under the name of *Oleum Origani Gallicum* and *O. O. Cretici;* it is always poor in carvacrol, and sometimes entirely free from it. It is, therefore, probably common origanum oil.

Cretic origanum oil is used, especially in Italy, as a remedy for carious teeth and for paralysis. It is frequently adulterated with oil of turpentine and copaiva oil.

*Marjoram oil* (sweet), *oleum marjoranæ* (*Marjoranöl*, G., *essence de marjolaine ou d'origan*, F.). Sweet marjoram, *Origanum Marjorana*, L., is an annual herb, indigenous to Western Asia and South-eastern Europe, and frequently cultivated for culinary purposes. By distilling the flowering herb in a fresh or freshly dried state, 0.3 to 0.9 per cent. of volatile oil is obtained. The oil from the dried herb is pale yellow, that from the fresh herb greenish-yellow. Rectified oil is colorless, but becomes dark by age. It has a pungent smell and a hot, peppery, and slightly bitter taste. Its specific gravity is 0.89 to 0.90 at 59° F., optical rotation + 17° 10′. The oil is limpid, but when badly kept, becomes thick and sticky, and occasionally deposits stearoptene, the latter taking place also when the oil is cooled to 6.8° F. It has an acid reaction. It dissolves in every proportion in an equal volume of 90 per cent. alcohol.

As regards the composition of marjoram oil it can only be said that it consists of a small quantity of a terpene and considerable quantities of an oxygenated body. The latter is perhaps a mixture of camphor and borneol, but probably a sesquiterpene hydrate, $C_{15}H_{24},H_2O$.

With iodine marjoram oil detonates; by nitric acid it is colored brown red and, on heating, converted into a brown-yellow balsam. Sulphuric acid is colored sanders-red, while the oil separates with a blood-red color, remaining unchanged as regards its consistency.

Marjoram oil is adulterated with oil of turpentine, petroleum, alcohol and thyme oil. It is used itself as an adulterant of peppermint oils. In England and France it is largely used for perfuming soap and also in the preparation of liqueurs.

*Savory oil* (*Bohnenkraut- or Saturejaöl*, G.) Both the summer savory, *Satureja hortensis*, L., and the winter savory, *S. montana*, yield by distillation with steam a volatile oil, which has recently been examined.

E. Jahns has investigated the *oil of summer savory*. The oil prepared from the dried herb was yellow, limpid, of an aromatic thyme-like odor, and specific gravity 0.898 at 59° F. The alcoholic solution of the oil was colored green by chloride of iron. By shaking the oil with soda lye two layers were obtained, an upper one consisting of hydrocarbons, and a lower watery alkaline one containing phenols; the latter separated in the form of a brown oil after acidulation with hydrochloric acid. They were redissolved in an equal quantity of 15 per cent. soda lye and the solution shaken with ether, whereby carvacrol passed into solution. The quantity of carvacrol, $C_{10}H_{14}O$, amounted to about 30 per cent. of the sweet savory oil. It had a specific gravity 0.981 at 59° F., and boiled between 449.6° and 451.4° F. (corrected between 456.8° and 460.4° F.) Its alcoholic solution was colored green by chloride of iron. The carvacrol was perfectly identical with that from Cretic origanum oil. A second phenol (only about 0.1 per cent. of the oil) remained behind in the soda lye, but could not be closer examined; with chloride of iron it gave a violet coloration and appeared to possess the same boiling point as the carvacrol. The portion of the oil not attacked by the soda lye began to boil at 343.4° F.; up to 40 per cent. of it passed over between 343.4° and 356° F., 50 per cent. between 356° and 374° F., and later on 4 per cent. more. It left behind a brown tarry residue and contained cymol and a terpene. By fractionating the distillate two principal portions were obtained, one boiling between 343.4° and 347° F., and consisting of cymol, $C_{10}H_{14}$; and the other boiling between 352.4° and 356° F., and consisting of a terpene, $C_{10}H_{16}$, which had a specific gravity of 0.855 at 59° F. The small quantity which passed over up to 374° F. was also a terpene. Hence the oil consists of 30 per cent. carvacrol, 20 per cent. cymol, and 50 per cent. terpene (boiling point 352.4° to 356° F.), besides traces of a second phenol.

Commercial oil from Southern France contained 10 per cent. cravacrol, 0.8 per cent. of the second phenol, cymol, terpene, and a not phenol-like oxygenated constituent. Its alcoholic solution was colored violet by chloride of iron.

The oil of *Satureja montana* or *winter savory*, has been described by A. Haller. The plant grows near Grasse and yields a not very limpid orange-yellow oil of an aromatic taste, reminding one of origanum oil, and of specific gravity 0.7394 at 62.6° F. The examination of the oil was carried out in the same manner as above described. By rectification, the phenol-mixture yielded a liquid body boiling between 449.6° and 451.4° F., which smelled of carbolic acid, had a specific gravity of 0.972 at 62.6° F., and consisted of carvacrol, $C_{10}H_{14}O$, identical with caraway carvacrol. By chloride of iron, its alcoholic solution was colored first green, then yellow. The oil contained 35 to 40 per cent. carvacrol. A second phenol, boiling at 482° F., also appeared to be present. The hydrocarbons of the oil yielded two fractions, one between 341.6° and 347° F., the other between 356° and 365°F., and appeared to be terpenes. Hence the two oils of *Satureja* agree in all principal points, though that of *S. montana* seems to be somewhat richer in carvacrol.

### *Verbenaceæ.*

*Verbena oil.* To the family of *Verbenaceæ* belongs a series of plants of the species *verbena*, which are distinguished partly by their fragrance and partly by their beautiful flowers. *Verbena triphylla*, Herit, which is indigenous to Peru, and whose leaves by rubbing diffuse an excellent odor, similar to that of lemon grass, yields by distillation with water the *genuine verbena oil.* Regarding its constitution, nothing definite is known, because lemon-grass oil is very frequently designated by the name of verbena oil. According to Gladstone, verbena oil has a red color and by distillation yields an oxygenated oil, while the resinified portion remaining behind evolves on heating sulphuretted hydrogen. According to Williams, verbena oil has a specific gravity of 0.895 to 0.896 at 60° F., boils at 429.8° F., absorbs 248 to 268 per cent. iodine and 1.2 to 1.37 per cent. caustic potash. The commercial essences and extracts of verbena do not contain verbena oil, but lemon-grass oil.

## X. *Volatile Oils of the Families Caprifoliaceæ, Valerianaceæ, Compositæ, Santalaceæ and Aristolochiaceæ.*

Of the oils belonging to this group none is of prominent technical importance, but most of them are of special scientific interest. The oils are obtained partly from the roots, partly from the flowers, partly from the flowering herb, and occur ready-formed in the plants. Only sandal-wood oil belongs to the fragrant oils. Most of the oils to be mentioned here are chiefly of medicinal importance, though partly also for the manufacture of liqueurs. With the exception of the oils of arnica and asarum, they are specifically lighter than water, and, with the exception of erigeron oil, their chief constituent is an oxygenated body, $C_{10}H_{16}O$, forming the chief constituent of chamomile oil (camillol), of feverfew oil (matricaria camphor), of wormwood oil (absinthol), of tansy oil (alantol). A body, $C_{10}H_{18}O$, forms the principal constituent in valerian oil (borneol) and in wormseed oil (cineol). Esters form essential constituents of the oils of valerian and Roman chamomile, and ethers of the oils of arnica and asarum, and partly also of valerian oil. In terragon oil occurs anethol, $C_{10}H_{12}O$; in sandalwood oil, santalol, $C_{15}H_{24}O$, and in elecampane oil, in addition to alantol, alantic acid, $C_{15}H_{20}O_2$. Hydrocarbon, in the form of limonene, occurs as principal constituent only in erigeron oil, though pinene, dipentene and other terpenes are also present in the oils of valerian, chamomile, feverfew, wormseed, tarragon, wormwood, tansy and asarum.

### *Caprifoliaceæ.*

*Elder oil, oleum sambuci* (*Hollunderöl*, G.; *essence de sureau*, F.). The flowers of *Sambucus nigra*, L., or the common elder, contain a small quantity of oil, which is obtained in the following manner: The oil is dissolved by distilling the fresh flowers with water. To saturate the aromatic water thus obtained more thoroughly with oil, it is again used in the distillation of a fresh charge of flowers, and finally shaken with petroleum-ether for the extraction of the oil.

As it is difficult thoroughly to shake large quantities of the aromatic water with petroleum-ether, it is best to use a simple mechanical contrivance, for instance a sheet-iron cylinder revolving around a shaft. The cylinder is filled three-quarters full with aromatic water and petroleum-ether, and slowly revolved, which effects an intimate mixture of the two liquids, and the volatile oil is in a comparatively short time completely absorbed by the petroleum-ether.

Pure elder oil is greenish or yellowish-white, crystalline, of butyraceous consistency and of a stupefying odor. According to J. H. Gladstone, elder oil yields by distillation first a small quantity of water and sulphuretted hydrogen, when a terpene $C_{10}H_{16}$ distils off. The residue consists of a white body crystallizing from ether; it is sparingly soluble in alcohol and insoluble in alkaline liquids.

Elder oil is used in perfumery, chiefly in combination with other volatile oils. Elder-flower water is also occasionally employed. It is prepared as follows: Distil 10 lbs. of freshly gathered flowers with 18 quarts of water until 10 to 13 quarts have passed over, and mix the distillate with about 3½ ozs. of strong alcohol. Elder-flower water is used in the preparation of elder milk, elder cold cream, etc. (Hirzel, Piesse).

### *Valerianaceæ.*

*Valerian oil, oleum valerianæ* (*Baldrianöl*, G.; *essence de valeriane*, F.). *Valerian, Valeriana officinalis*, L., is a native of Europe, from the Mediterranean northward, and of Northern Asia, and is cultivated to some extent in Holland and England, and in this country, in New England and New York. The volatile oil is obtained by distilling the crushed root, which, however, should not be too old, with steam. The yield varies between ½ and 2 per cent., and is largest from valerian in dry localities.

Valerian oil from the fresh root is grass green, and also that from valerian grown in a forest, while the oil from old dried root and from valerian grown in swamps is yellow to brown. Fresh oil is limpid, but becomes thicker and darker by age. The odor of the oil resembles that of valerian, but is more penetrating and more disagreeable. Its taste is camphor-like, aromatic, disagreeable, but not pungent. Its specific gravity is 0.936 at 50° F., and increases with age, that of the oil of drug stores being generally 0.93 to 0.96. Its boiling point is given at from 248° to 320° to 772° F. At 5° F. it separates valerianic acid in white flakes, and on account of this content of acid has an acid reaction. It does not solidify even at

—40° F. In alcohol it is readily soluble. It turns the polarized ray of light to the left (—15.5°). By reason of its content of valerianic acid, the water passing over with the oil is very acid.

Valerian oil consists of 25 per cent. pinene, $C_{10}H_{16}$, 5 per cent. acids, especially valerianic acid, $C_4H_9COOH$, a liquid alcohol, $C_{10}H_{18}O$, esters of borneol with bornyl formiate, bornyl acetate and borynl iso-valerianate.

A magnificent blue color is produced when one drop of the oil, dissolved in fifteen drops of carbon disulphide, is well shaken with sulphuric acid, and afterwards one drop of nitric acid of specific gravity 1.2 is added.

Valerian oil is used, to a limited extent, in medicine; it has been employed with advantage in hysteria, chorea, and even in epilepsy.

*Kesso root oil* or *Japanese valerian oil.* The Japanese valerian root is derived from *Valeriana officinalis*, L., *var. augustifolia*, and not from *Patrina scabiosaefolia*, Link., as was formerly supposed. The root yields 6 to 6.5 per cent. of oil. The latter has a greenish color, is somewhat thickish, and as regards odor and taste can scarcely be distinguished from ordinary valerian oil except that the odor is more powerful and its aroma more lasting. Its specific gravity is 0.996 at 59° F., and it boils between 338° and 581° F. Its known constituents are pinene, dipentene, terpineol, borneol, bornyl acetate, bornyl iso-valerianate, and kessyl-acetate. Kesso-root oil is used for the same purpose as ordinary valerian oil.

### *Compositæ.*

*Chamomile oil*, (*Roman*), *oleum chamomillæ Romanæ* (*Römisch-kamillenöl*, G.; *essence de camomille Romaine*, F.). The Roman or English chamomile, *Anthemis nobilis*, L., is indigenous to southern and western Europe as far north as England, and cultivated in Germany, Great Britain, France and Belgium. The oil is obtained by distilling the freshly dried flowers with steam. The yield is from 0.6 to 0.9 per cent., and appears to be larger from plants grown in the north.

Roman chamomile oil obtained from fresh flowers is intensely blue and retains this color for quite a long time. The oil from dried flowers is also blue, but soon turns to greenish-yellow or greenish-white, which is the color of commercial oil. It has a strong, agreeable odor, a pungent taste, and an acid reaction. Its specific gravity ranges from 0.905 to 0.915 at 59° F. It commences to boil at 320° F., the boiling point rising to 374° F., where it remains stationary for some time, the temperature reaching 410° F. only towards the end.

On account of its numerous, largely non-aromatic constituents, Roman chamomile oil is very interesting in a chemical respect. In the fraction between 296.6° and 298.4° F., it contains, in addition to an unknown hydrocarbon, isobutyric acid isobutyl-ester, $C_3H_7COOC_4H_9$; in the fraction between 350.6° and 352.4° F., angelic acid isobutyl ester, $C_4H_5COOC_4H_9$; in the fraction between 392° and 393.8° F. angelic acid amyl ester, $C_4H_7COOC_6H_{11}$; in the fraction between 399.2° and 401° F., tiglic acid amyl ester, $C_4H_7COOC_5H_{11}$; further methacrylic acid, $C_2H_2(CH_3)COOH$, in the form of an ester; finally the angelic acid and tiglic acid esters of a hexyl alcohol, $C_6H_{13}OH$, and of anthemol, $C_{10}H_{15}OH$, and probably also free angelic acid, $C_4H_7COOH$. Roman chamomile oil is so rich in combinations of angelic and tiglic acids that both acids, but particularly the former, may be obtained from it. Both acids are isomeric; tiglic acid is $CH_3CH = C(CH_3)COOH$, and angelic acid probably $CH_2 = C(CH_3)CH_2COOH$. The principal mass (about 70 per cent.) is formed of the fraction between 302° and 392° F., only 17 per cent. passing over between 392° and 482° F.

*Chamomile oil*, (*common*), *oleum chamomillæ vulgaris* (*Kamillenöl*, G., *essence de camomille commune*, F.). The German chamomile, *Matricaria Chamomilla*, L., is common in fields and waste places throughout Europe as far as Finland, and in the temperate parts of Asia; it has been completely naturalized in Australia, and is occasionally cultivated in German settlements in the United States. The dried flower-heads possess a peculiar, somewhat disagreeable odor. They contain 0.13 to 0.3 per cent. of volatile oil, the greatest yield and best quality of oil being obtained by distilling the freshly dried flowers with steam. Salted flowers also are said to yield a good quality of oil. In distilling, metal Florentine flasks should be used, as the oil adheres tenaciously to glass vessels, and the distillate has to be treated with ether. Care must also be taken that an oil floating upon the water which passes over in distilling, is not mixed with the specifically heavier portion of the distillate, the actual chamomile oil.

Chamomile oil is of a beautiful, dark blue color, and almost opaque. It is thickly-fluid, thick at 32° F. and solid at 21.2° F. Its specific gravity ranges from 0.930 to 0.945 at 59° F. It begins to boil at 221° F.; at above 491 °F., it forms a beautiful blue vapor and up to 572° F., distils almost entirely off. It possesses a penetrating odor of chamomile, and an aromatic chamomile-like taste. To preserve its blue color it has to be kept excluded from light and heat, otherwise it becomes green, and finally brownish and viscous. It is readily soluble in alcohol and has a neutral reaction.

Chamomile oil consists of camillol, $C_{10}H_{18}O$, an unknown terpene $C_{10}H_{16}$, and the "blue oil" which very likely is tricamillol, $C_{30}H_{48}O_3$.

Chamomile oil frequently consists only of another volatile oil (oil of turpentine or lemon), distilled over chamomile flowers. Genuine oil is also adulterated with oils of copaiva and cedarwood, and with alcohol. Such sophistications may be recognized by the behavior of the oil toward iodine, with which chamomile oil does not give a violent reaction, as well as by its behavior toward common salt solutions, from which genuine oil separates almost unchanged. However, according to Weppen and Lüders, an adulteration of chamomile oil with millefoil oil can be scarcely detected. Chamomile oil is used in medicine, internally as well as externally, for asthma, colic, spasms in the stomach, whooping cough, etc.

*Feverfew oil* (*Mutterkrautöl*, G., *essence de matricaire*, F.)

The stems, leaves and flowers of the feverfew, *Chrysanthemum Parthenium*, B., *Matricaria Parthenium* L., *Pyrethrum Parthenium*, Sm., yield by distillation a volatile oil, that from the fresh herb being greenish, and that from the dried herb straw-yellow. The yield is small, being not more than 0.1 to 0.4 per cent.

According to the investigations of J. Chautard, feverfew oil contains a terpene, $C_{10}H_{16}$, which, however, could not be obtained in a pure state. It seems to resemble oil of turpentine and boils below 392° F. It is levo-rotatory and forms a levo-rotatory monohydrochloride. The oil further contains an oxygenous body which boils at over 428° F. and contains more oxygen than the matricaria camphor; it is dextro-rotatory and occurs in varying quantities in the oil. Matricaria camphor is obtained by cooling the oil, and another portion of it by treating the non-solidified part with nitric acid. The camphor is colorless, transparent, sublimes in octahedrons, has a specific gravity of 0.9853 at 64.4° F., melts at 347° F., and boils at 399.2° F. Small pieces of it thrown upon water show the revolving motion of ordinary camphor. The alcoholic solution of the camphor is levo-rotatory to the same degree as a corresponding solution of laurel camphor is dextro-rotatory. The terpene as well as the camphor occur chiefly in the leaves, the "oxidized oil" in the flowers. According to Chautard, matricaria camphor might serve as a substitute for ordinary camphor.

*Wormseed oil*, *oleum cinæ* (*Wurmsamenöl*, G.; *essence de semen-contra, de semencine ou de barbotine*, F.). The so-called wormseed consists of the unexpanded flower-heads of several species of *Artemisia*, especially of *A. maritima*, L., also called *A. Cina*, indigenous to the Orient and Southern Russia. Levant wormseed is considered the best, while the Barbary or African and the East Indian varieties are not allowed to be used officinally. Wormseed has a strong, disagreeable, aromatic odor, and a bitter, camphoraceous, disagreeable taste. The effective constituent of the seed is *santonin*, $C_{15}H_{18}O_3$, which in larger doses is poisonous, but in smaller doses the best anthelmintic known. Santonin being odorless and almost tasteless, the odor and taste of wormseed must be due to the volatile oil contained in it. Santonin is obtained by several times boiling the bruised wormseed with water, and by distilling off the oil. However, the oil thus obtained possesses different properties from that produced by distilling the wormseed with steam (*i. e.*, without the use of lime). This fact may explain the contradictory statements regarding the properties and composition of wormseed oil. Barbary wormseed is richer in volatile oil than Levant seed.

Wormseed oil derived from santonin factories, which is the product mostly found in commerce, is thickly-fluid and dark-yellow, while the oil obtained by distillation without lime is quite limpid and pale yellow. Of the oil from santonin factories about 53 per cent. passes over between 347° and 356° F., 32 per cent. between 356° and 365° F., and 11 per cent. more up to 401° F. (Wallach). At 428° F. only a very small residue remains behind (Völkel). The specific gravity of this oil, according to Hirzel, is 0.945 at 46.4° F., and according to Wallach, 0.92715 at 60.8° F. The pale yellow oil obtained by distilling the seed with steam has, according to Völkel, a specific gravity of 0.936, according to Hirzel, of 0.946 at 51.8° F., and boils mainly between 347° and 356° F. Both oils become colorless by rectification, the former, however, with greater difficulty. The odor of the crude oil is penetrating and disagreeable like that of the seed, and its taste bitter camphoraceous. Fresh oil has a neutral reaction, while old oil is yellow-brown and shows an acid reaction. In water the oil is sparingly soluble, but in every proportion in alcohol of specific gravity 0.85.

Wormseed oil was examined by Völker and, according to him, is a mixture of various oxygenated compounds, of which Hirzel distinguished cinæbene, cinæbene-camphor and others. However, for a thorough explanation of the composition of wormseed oil we are indebted to the investigations of O. Wallach and W. Brass. Below 356° F. the oil yielded a very small quantity of first running, which smelled of aceton and reduced nitrate of silver. The principal mass of the oil, after rectification, boiled between 348.8° and 352.4° F., had a specific gravity of 0.92067 at 60.8° F. and possessed the composition $C_{10}H_{18}O$. By the introduction of HCl and simultaneous cooling pure cineol, $C_{10}H_{18}O$, could be obtained from it. From the higher fractions a portion boiling at 422.6° F. was selected, the analysis of which led to the formula $C_{15}H_{26}O$. Hence the principal fraction contains cineol and probably a hydrocarbon, boiling at nearly the same temperature; while the higher boiling fractions contain a body poorer in carbon and richer in oxygen than cineol. The property of cineol has previously been referred to, but it may be again mentioned here, that under the influence of various reagents it yields cinene, $C_{10}H_{16}$ = dipentene.

Hence worm-seed oil consists mainly of cineol, $C_{10}H_{18}O$, mixed with a terpene and a combination richer in oxygen, and perhaps with combinations of propionic acid or aceton.

Wormseed oil is used, to a limited extent, in medicine.

*Mugwort oil* (*Beifussöl*, G.). The herb as well as the root of the mugwort, *Artemisia vulgaris*, L., contains volatile oil. The plant grows spontaneously in the United States, but is also cultivated. The root, *Radix Artemisiæ*, in a powdered state, is medicinally employed in Europe. For the production of oil the entire herb may be used, but the root contains oil only in the outer layer.

Mugwort oil is pale greenish-yellow, butyraceous, of a penetrating odor, and bitterish burning, afterwards cooling taste. It has a neutral reaction. The yield from the herb is 0.2 per cent. and from the root 0.1 per cent. The specific gravity is 0.920 at 59° F.

*Alpine mugwort oil* from the herb of *Artemisia glacialis*, Luc., possesses a strong aromatic odor, and is suitable for the better qualities of Benedictine and Chartreuse liqueurs. The yield is 0.25 per cent. The oil has a specific gravity of 0.964 at 59° F., and boils between 383° and 410° F.

*Tarragon oil* (*Esdragonöl*, G.) Tarragon, *Artemisia Dracunculus*, L., is indigenous to Southern Europe and Siberia, and is frequently cultivated. By distilling the herb, especially the leaves, with steam a pale greenish oil of a peculiar odor and mild aromatic taste is obtained. It has a specific gravity of 0.94 to 0.95, and boils between 392° and 402.8° F. It dissolves readily in 90 per cent. alcohol and chemically resembles aniseed oil. According to Laurent, the portion passing over at 402.8° F. forms a colorless fluid of specific gravity 0.945 at 59° F., which mixes with ether and dissolves in an equal volume of alcohol. In the air the fraction is constant, is not changed by caustic potash, but is resinified by fuming sulphuric acid. The formula of tarragon oil is $C_{32}H_{42}O$. The principal constituent of the oil is anethol, $C_{10}H_{12}O = C_6H_4C_3H_5OCH_3$. In addition it contains a small quantity of a hydrocarbon, which is more volatile than anethol and passes over below 402.8° F. By the introduction of chlorine and simultaneous heating, tarragon oil is converted into a thick oil from which dragonyl chloride, $C_{32}H_{32}Cl_{14}O_3$ · (Laurent) or $C_{20}H_{10}Cl_8O_2$ (Gerhardt) may be isolated. It forms a turpentine-like mass, and by treatment with alcoholic potash lye yields chlorodragonyl $C_{32}H_{33}Cl_{12}O_2$ (Laurent), a thick oily fluid.

Tarragon oil is chiefly used for scenting vinegar.

*Wormwood oil*, *oleum absinthii*, (*Wermutöl*, G., *essence d'absinthe*, F.). Wormwood, *Artemisia Absynthium*, L., is a perennial plant indigenous to hilly and mountainous regions of Northern Africa, the greater portion of Europe, and the northern part of Asia. It is frequently cultivated for medicinal purposes, both in Europe and in this country, where it has become naturalized in some localities, and grows wild in waste places and along roadsides. The herb has a very spicy odor and sharp, aromatic, very bitter taste. Its essential constituent is a bitter principle, *absinthin*, $C_{10}H_{56}O_8 + H_2O$; in addition it contains a volatile oil. Several varieties of wormwood, which grow in the Alps, especially *A. Mutellina*, Vill., *A. glacialis*, L., and *A. Spicata*, Wulf. contain aromatic constituents, are less bitter than actual wormwood, and serve chiefly for the preparation of the liqueur *extrait d'absinthe*. For obtaining the oil it is best to use the fresh flowering plant; the dried herb, if used, has to be moistened and allowed to lie several hours previous to distillation. The average yield, according to Maier, is 0.87 per cent., while Schimmel & Co., give it as 0.2 to 0.4 per cent. The leaves are richest in oil and yield a limpid oil, while that from the flowers and seed is thickly-fluid. Wormwood grown in the north is richer in oil than the herb derived from the south. In commerce, French and American oils are considered the best qualities.

Wormwood oil from fresh herb is green, that from dried herb greenish to yellow-brown, but with age every kind of wormwood oil becomes opaque, dark brown and somewhat viscous, while fresh oil is limpid. Its odor is strong, somewhat disagreeable of the plant, and its taste bitter and acrid. Its specific gravity ranges from 0.877 to 0.94, the oil from the flowers and seed having the higher specific gravity. It boils in the main between 356° and 401° F., whereby it becomes more and more colored, and finally yields also colored distillates. The oil has a neutral reaction, is dextro-rotatory, and dissolves readily in alcohol.

Wormseed oil consists preponderatingly of absinthol, $C_{10}H_{16}O$ (boiling point 383° to 393.8°F.), mixed with a small quantity of terpene, $C_{10}H_{16}$ (boiling point 302° F.), and hydrocarbon (boiling point 338° to 356° F.), as well as blue oil.

Iodine dissolves in fresh oil to a thick, green, later on dark brown liquid, without heating to any extent. If, however, the oil has for some days been in contact with air, the iodine is taken up with an explosive reaction and heating, whereby a viscous, green residue is formed.

Wormseed oil is adulterated with alcohol, copaiva oil and turpentine oil. Oil thus adulterated becomes readily pale yellow and resinifies with ease; its odor is also changed. Formerly a kind of wormwood oil was produced by distilling turpentine oil over the herb provided with nearly ripe seed.

Wormwood oil has a tonic and exciting effect upon the digestive organs, and in this sense is used internally as well as externally for embrocations. It is also a constituent of the well-known liqueur absinthe.

*Milfoil oil* (*Schafgarbenöl*, G.). Milfoil, *Achillea millefolium*, L., is a perennial herb, growing in fields and pastures throughout the greater part of the northern temperate zone of America and Europe. The herb has an aromatic odor and a tart, bitterish taste. The flowers have a bitter taste and a peculiar, aromatic odor, contain a bitter principle and a volatile oil, both occurring also to a smaller extent in the leaves. A few other varieties of milfoil grow in the Alps, for instance, *A. moschata*, Wlff., and *A. strata*, L., the aromatic herb of which forms a constituent of the so-called Swiss tea. From the *Achillea moschata* of the Alps the iva liqueur is prepared. *Achillea nobilis*, L., and *A. ageratum*, L., also contain volatile oils, which, however, do not possess the same properties.

*Milfoil oil* (*common*), *from Achillea millefolium*, L. The fresh flowers yield on an average 0.147 per cent. of oil; dried flowers, 0.19 per cent., and dried herb, 0.09 per cent., but, according to Schimmel & Co., 0.8 per cent. The color of the oil appears to be dependent on the locality where the plant is grown, it varying between yellow, green and blue. Old oil is yellow or brown. The oil has a penetrating, milfoil-like odor, and an aromatic, camphoraceous taste. It is viscous, in the cold butyraceous, and inclined towards resinification. Fresh oil has a neutral, and old oil an acid, reaction. Its specific gravity ranges from 0.85 to 0.92. In water it is sparingly soluble, but readily in alcohol. Iodine is absorbed under heating: nitric acid colors the oil reddish-brown, and, on heating, resinifies it; sulphuric acid colors it reddish-brown, and, on heating, thickens it.

*Milfoil oil, from Achillea nobilis*, L. The yield is, on an average, 0.25 per cent. The oil is pale yellow, viscous, of a strong, agreeable odor, and camphoraceous taste, and has a specific gravity of 0.97 to 0.98.

*Iva oil, from Achillea moschata*, D. C., is a bluish, green oil of penetrating odor and peppermint-like taste. Its specific gravity is 0.934 at 59° F., and it boils between 338° and 500° F. According to v. Planta, its chief constituent is ivaol, $C_{12}H_{20}O$, which boils between 338° and 410° F. In addition the oil contains a resin.

*Milfoil oil, from Achillea ageratum*, L. This plant is frequently met with in the gardens of Northern Italy, where it is cultivated on account of its beautiful yellow flowers. When rubbed between the hands, the plant diffuses an aromatic camphoraceous odor. Before and during flowering the plant when distilled with water yields a volatile oil which has been examined by S. de Luca. The plant is richest in oil shortly before flowering; later on the quantity of oil decreases, and the oil becomes richer in resin and volatilizes with greater difficulty. The specific gravity of the oil is 0.849 at 75.2° F. The first distillate passes over between 329° and 338° F., and another between 356° and 359.6° F. The second fraction, as regards composition, corresponds to the formula $C_{26}H_{44}O_3$. The first fraction remains liquid at 0.40° F.; by chlorine it is colored reddish, with heating, and acquires a copal-like odor; bromine acts still more energetically upon the fraction. The oil is constant in the air, but is rendered turbid by bisulphate of soda.

*Milfoil root oil*, from the root of *Achillea millefolium*, L. The yield is only 0.032 per cent. The oil is slightly yellowish and possesses a disagreeable valerian-like odor.

*Erigeron oil* (*Erigeronöl* G.) is obtained from *Erigeron Canadense*, L., known throughout North America as horse-weed, mare's tail, fireweed, butterweed, colt's tail, etc. It flourishes in fields and waste places, and has established itself as a more or less common weed in most countries. Pure erigeron oil has, according to F. Power, a specific gravity of 0.8498, is colorless and neutral, possesses an agreeable odor, and boils at 348.8° F. It is a terpene, $C_{10}H_{16}$, which according to F. Beilstein and E. Wiegand, boils at 348.8° F., and has a specific gravity of 0.8468 at 64.4° F. It forms a crystallized dihydrochloride, which melts at 116.6° to 118.4° F. The terpene may be considered limonene, which has also been confirmed by O. Wallach. The latter prepared from the terpene the tetrabromide melting at 219.2° to 221° F. According to A. Todd, erigeron oil is dextro-rotatory, but in a rectified state almost inactive. The first fractions are dextro-rotatory. The oil, when rectified and free from resin, has a specific gravity of 0.855 to 0.865. It begins to boil at 342° F. and at least 5 per cent. distils off up to 347° F. When erigeron oil is distilled in a current of steam a dark reddish-brown resin remains behind, while the distillate is perfectly colorless.

*Tansy oil, oleum tanaceti* (*Rainfarnöl*, G., *essence de tanaisie*, F.) Tansy, *Tanacetum vulgare*, L. is a perennial herb indigenous to Europe and Central Asia, and naturalized in many parts of North America, where it grows in old fields, along roadsides, and in the neighborhood of river banks. According to O. Leppig, the flowers of the plant contain 1.49 per cent. of oil, but the herb only 0.66 per cent. Fresh flowers yield more oil than fresh leaves, and fresh plants more than dried. The yield by distilling the fresh flowering herb with water is 0.104 to 0.416 per cent.; according to Schimmel & Co., 0.15 to 0.25.

Tansy oil is, according to G. Bruylants, a mobile, yellowish liquid becoming brown on exposure to air and light. Its odor is strong, reminding one of camphor. Its specific gravity is 0.923 at 59° F. About 90 per cent. of the oil distils off between 377.6° and 536° F., about 10 per cent. of residue remaining behind. According to Maier, oil from plants grown upon a moist soil has a greenish color. The odor is penetrating and disagreeable, and the taste bitter, sharp, and pungent. The oil has a slightly acid reaction, and is readily soluble in alcohol.

The composition of tansy oil has been ascertained by G. Bruylants. By shaking the oil with concentrated solution of $NaHSO_3$ and precipitating with alcohol he obtained a crystalline separation of the composition $C_{10}H_{15}NaSO_3$; by heating it with dilute soda lye a colorless oil passed over, which possessed only a slight odor of tansy, had a specific gravity of 0.918 at 39.2° F., remained liquid at 6.8° F., and boiled at 383° to 384.8° F. Analysis led to the formula $C_{10}H_{16}O$, and the substance received the name *tanacetylhydrine*. It showed the properties of ordinary camphor, combined, however, directly with hydrogen and acid sodium sulphide, and behaved like an aldehyde towards silver nitrate solution. After the separation of the sodium sulphide combination, an oil commencing to boil at 338° F. and distilling off at 446° F. may be obtained from the liquid portion. From this oil a fraction was isolated between 311° and 320° F., which contained a terpene, $C_{10}H_{16}$. The fraction between 397.4° and 401° F., however, has the formula $C_{10}H_{18}O$ and is tanacetyl alcohol. This alcohol, by oxidation with chromic acid, is converted into ordinary camphor; by distillation over $P_2O_5$ it yields a terpene boiling at 320° to 329° F. Hence, Bruylants found in tansy oil 1 per cent. terpene, 70 per cent. tanacetylhydrine, and 26 per cent. tanacetyl alcohol. According to F. D. Dodge, tanacetol and tanacetyl alcohol belong to the same group as citronella aldehyde, geraniol, etc. In the flowering herb of the tansy plant occurs, according to O. Leppig, in addition to other acids diffused in the vegetable kingdom, *malic acid*, as well as tannin and the bitter principle *tanacetene*. To the malic acid is probably due the frequently observed acid reaction of the oil. Finally it may be remarked that Roscoe applies to tanacetylhydrine the name of tanacetol.

According to H. Peyraud, tansy oil has a poisonous effect, it acting partly similar to strychnine and partly like camphor and wormwood oil. Tansy oil appears to be chiefly used in the form of *eau de tanaisie* for cosmetic purposes.

*Arnica oil, oleum arnicæ* (*Wohlverleih-or Arnikaöl*, G.; *essence d'arnica*, F.). The arnica plant, *Arnica montana*, L., is indigenous to Europe, growing upon the mountains of Switzerland and Germany, and further north in the plains. It is likewise met with in Northern Asia and in the south-western part of America. Both the root and the flowers are officinal. The root has a disagreeable aromatic somewhat musty odor, and a sharp, bitterish taste; it contains volatile oil, and the bitter principle *arnicin*, $C_{20}H_{30}O_4$. The arnica flowers have a sharp and bitter taste, and a peculiar, not disagreeable odor; they are richer in arnicin than the roots, and also contain a volatile oil. For the purpose of obtaining the volatile oil the roots or flowers are moistened with water, allowed to lie for some time, and then subjected to distillation with water or steam. The roots yield 0.4 to 1.5 per cent. of oil, according to their age, the yield from roots gathered in the fall being best. Fresh flowers yield 0.028 per cent., and dried flowers 0.007 per cent. of volatile oil.

A distinction has to be made between arnica flower oil and arnica root oil. *Arnica flower oil* is blue, bluish-green, green or generally yellowish, but becomes brown by age. It has a strong, spicy odor and taste, reminding one somewhat of chamomile oil. It is viscous and even butyraceous, and has a specific gravity of 0.9. In alcohol it is soluble with great difficulty, it dissolving only in 16 to 20 parts of absolute alcohol, or in 100 parts of alcohol of specific gravity 0.85. Fresh oil is neutral, while older oil has a slightly acid reaction.

*Arnica root oil*, the ordinary oil of commerce, is yellow, very slightly greenish, and becomes somewhat darker by age. It has a strong odor of the root, and a pungent, aromatic taste. It is not very thickly-fluid and is readily soluble in ether and alcohol. Its specific gravity ranges from 0.990 to 1 at 59° F. It has a neutral reaction.

Only arnica root oil has been subjected to thorough chemical investigations, which show that the oil contains about 20 per cent. isobutyric acid phlorol-ester, $C_3H_7COOC_8H_9$, and 80 per cent. thymohydroquinone-methyl ether, $C_{10}H_{12}(OCH_3)_2$, mixed with a small quantity of phlorol-methyl ether, $C_8H_9OCH_3$. It may be remarked that thymo-hydroquinone corresponds to the formula $C_3H_7C_6H_2(OH)_2CH_3$ and, hence, the principal constituent of arnica root oil is $C_3H_7C_6H_2(OCH_3)_2CH_3$, or dimethylthymo-hydroquinone. Furthermore ordinary phenol is identical with $\beta$-ethyl-phenol, $C_8H_4C_2H_5OH$. Now since thymol belongs to the meta series and a derivative of it occurs in arnica oil, it is possible that the phlorol of arnica oil is also meta-ethyl phenol. (Roscoe.)

The action of nitric acid may serve as a means of distinguishing arnica root oil from arnica flower oil, the latter, when heated with the acid, being converted into a reddish-yellow resin, while the former is colored grass-green, gas being evolved thereby.

Arnica oil is chiefly used in medicine.

*Elecampane oil* (*Alantol*, G.; *essence d'aunée*, F.). Elecampane, *Inula Helenium*, L., is a native of southern Asia and southern Siberia, and is found in southern Europe. It is not unfrequently cultivated. In the United States it grows spontaneously along roadsides and in pastures from New England south to the mountains of North Carolina and westward to Illinois. The thick, fleshy root is brought into commerce sliced either longitudinally or transversely. It contains a starch-like body called *inulin*, $C_6H_{10}O$; further alant-camphor or *helenin*, $C_6H_8O$ or $C_6H_{10}O$, and finally volatile oil, of which it yields 0.6 per cent. Alant camphor being the essential and effective constituent of the root, the oil is largely obtained as a by-product in the preparation of helenin. For the purpose of obtaining elecampane oil, the fresh roots collected in the fall are cut up and distilled with steam. Dried roots have to be coarsely powdered, moistened with water, and allowed to lie 12 to 18 hours before being subjected to distillation. According to J. Kallen, the distillate forms a white crystalline mass, consisting of a liquid and a solid portion. By pressing the mass the liquid portion is absorbed by filtering paper and can be obtained quite pure by subjecting the paper to distillation with water. The distillate forms a slightly yellowish liquid of an aromatic taste and peppermint-like odor, and boils at 392° F. The composition of the liquid corresponds to the formula $C_{10}H_{16}O$, the body being called *alantol*. By distillation over phosphorous pentoxide, alantol yields a hydrocarbon, $C_{10}H_{14}$, hence, cymol, boiling at 347° F.

The solid portion remaining behind in pressing off the alantol forms, after recrystallization from alcohol, colorless prismatic needles of a slight odor and taste, which melt at 150.8° F., boil at 527° F. (however, under partial decomposition), and sublime by slight heating. The substance has the formula $C_{15}H_{20}O_2$, and is alantic anhydride. When boiled with dilute potash lye potassium alantate is formed. Alantic acid has the formula $C_{15}H_{22}O_3$ and is a diatomic monobasic acid, probably $C_{14}H_{20}$-(OH)COOH. It forms fine needles, which are sparingly soluble in boiling water, but readily in alcohol; they melt at 194° to 195.8° F., whereby they are converted into anhydride.

On extracting elecampane root with alcohol and mixing the extract with water a crystalline substance melting at 161.6° F. is separated; this substance consists of a mixture of helenin, $C_6H_8O$, alantol, $C_{10}H_{16}O$, and alantic anhydride, $C_{15}H_{20}O_2$. The mixture of these substances was formerly called helenin or alant-camphor; the quantity of alantol contained in it is inconsiderable. According to Schimmel & Co., helenin consists of alantic anhydride, melts at 150.8° F., and has been successfully used in cholera.

According to Schimmel & Co. alantol, $C_{20}H_{32}O$ (?) is an aromatic levo-rotatory liquid which boils at 302° F., combines after a certain time with water, and is said to possess ozonizing effects. The mixture of alantic anhydride and alantol is said to have produced good results in the treatment of tuberculosis. Bufalini and Martini employ helenin in bronchial catarrhs and have observed no disadvantageous secondary effects. According to Mierzinski, elecampane oil is occasionally used in the preparation of liqueurs, and is sometimes adulterated with oil of turpentine or alcohol. It appears that these statements refer to alantol.

*Carline-thistle oil* (*Eberwurzelöl*, G.; *essence de chardon doré*, F.). The root of the carline thistle, *Carlina acaulis*, L., a species of thistle indigenous to Middle Europe, has a sweetish, aromatic taste and odor, and, in addition to resin and sugar, contains about 1 per cent. volatile oil. The oil has a dark red-brown color and a specific gravity of 1.033 to 1.036 at 59° F. By distillation about 50 per cent. of the oil passes over between 509° and 572° F., while the rest carbonizes; hence fractional distillation has to be effected in a rarefied space. Under a pressure of 22 mm. the first portions of the oil pass over at 289.4° F. Up to 334.4° F. a pale yellow distillate amounting to 40 per cent. of the oil was obtained, from which a hydrocarbon (16 per cent. of the oil) boiling at 289.4° to 311° F. and having a specific gravity of 0.9193 may be isolated. This hydrocarbon is very rich in carbon, but cannot be entirely separated from the admixed oxygenated substances. By heating the crude oil or the fraction 289.4° to 311° F. over sodium, a heavy evolution of gas takes place; by distillation a colorless oil passes over, the composition of which indicates the presence of a sesquiterpene, $C_{15}H_{24}$; it has a specific gravity of 0.8733 at 73° F. and the crude oil contains about 12 per cent. of it. However, the principal constituent of carline-thistle oil consists of a specifically heavier portion passing over between 336.2° and 339.8° F. under 21 mm. pressure, which, like a crystallizing body separating from the oil on exposure to cold, has not yet been closely examined.

### *Santalaceæ.*

*Sandal wood oil*, *oleum ligni santali* (*Santelholzöl*, G.; *essence de santal*, F.) is distilled from the wood of *Santalum album*, L., the white santal, a small tree indigenous to Southern India and some of the East Indian islands, and is cultivated in other parts of tropical Asia. The Mysore government, India, has long had establishments for extracting the oil, which is sold at annual auctions along with the wood, and chiefly bought up for exportation to China and Arabia. It is procured from the wood by distillation, the roots yielding the largest quantity and the finest quality of oil. The body of the still is a large globular clay pot, with a circular mouth, and is about 2½ feet deep by about 6 feet in circumference. No helmet is used, but the mouth of the still, when used, is closed with a clay lid, having a small hole in its centre, through which a bent copper tube about 5½ feet long is passed for the escape of the vapor. The lower end of the tube is conveyed inside a copper receiver, placed in a large porous vessel containing cold water. When preparing the sandal for distillation, the white or sap wood is rejected and the heart wood cut into small chips, of which about 50 pounds are put into the still. As much water is then added as will just cover the chips, and distillation is carried on slowly for ten days and nights, by which time the whole of the oil is extracted. As the water from time to time gets low in the still, fresh supplies are added from the heated contents of the refrigerator. The quantity of oil yielded by wood of good quality is about 20 ounces from 50 pounds.

According to Schimmel & Co., the principal East Indian markets are Telicherry and Calicut. The wood grown in the Dutch Indies is sold in Macassar; the oil obtained therefrom is less fine. Only about one-third of the wood sold in the East Indies goes to Europe, two-thirds of it being consumed in India itself.

The so-called *West Indian sandal-wood oil* is derived from Venezuela and is brought into commerce from Puerto Cabello. It is very likely derived from the wood of a Rutacea, and hence not identical with the genuine sandal-wood oil. It is of inferior quality and in little demand. According to Holmes, it is dextro-rotatory, while the East Indian oil is levo-rotatory.

East Indian sandal-wood oil has the consistency of thin syrup, is pale yellow to dark yellow and brown, possesses an agreeable rose-like odor, has a specific gravity of 0.963 to 0.967 at 59° F., and boils between 572° and 644° F.

According to the investigations of P. Chapoteaut, sandal-wood oil consists almost entirely of two oxygenated bodies, one of which boils at 572° F., and the other at 590° F., the body boiling at 572° being present in larger quantity. It has the formula $C_{15}H_{24}O$, and is called *santalal*. In character it resembles an aldehyde. By distilling it over phosphorous pentoxide the hydrocarbon, $C_{15}H_{22}$, which is probably identical with cedrene, passes over. The product from sandal-wood oil boiling at 590° F. has the formula $C_{15}H_{26}O$, and may be called *santalol;* it is an alcohol, and when treated with $P_2O_5$ yields *santalene*, $C_{15}H_{24}$, boiling at 500° F. If sandal-wood oil in a closed vessel be heated to 590° F., water and hydrogen are split off and the bodies $C_{20}H_{30}O$ (boils at 464° F.), $C_{40}H_{62}O_3$ (boils at 644°) and $C_{40}H_{60}O_2$ (boils at above 662° F.) are formed.

By concentrated sulphuric acid sandal-wood oil is colored, with strong heating, dark red to black. Nitric acid converts the oil into a yellow resin.

Sandal-wood oil is frequently adulterated with the oils of cedar, copaiva, etc., for the detection of which M. Durand gives the following tests: 1. Prepare a fresh ammonio-copper solution by saturating water of ammonia with black oxide of copper and filtering. Then mix 20 grammes of the filtrate with 80 grammes of water. Place 10 grammes of this solution in a test-tube and add 2 grammes of the oil. Shake forcibly. After 24 hours the contents of the tube will have formed a white and opaque soap. If the sandal oil contains any oil of cedar, even as little as $\frac{1}{10}$ of one per cent., the soap has a more or less green tint. 2. A mixture of 6 grammes bromine, 20 grammes 90 per cent. alcohol, and 64 grammes water, produces different reactions with the oils of cedar, copaiva, and sandal. The reaction is made with 1 gramme of the oil and 15 grammes of the solution. The precipitate formed with sandal oil looks like liquid honey, that with oil of cedar like tar, and that with oil of copaiva is olive green. By practice a mixture of these oils may be recognized to a fraction of 1 per cent.

Cripps* has examined a large number of samples of sandalwood oil and cedar-wood oil, and the result of this investigation is embodied by him in the following tests, to which he requires a good quality of East Indian sandal-wood oil to conform: The oil should be strongly levo-rotatory, its specific gravity may not be less than 0.970, it should give a clear solution with five times its volume of diluted alcohol (five parts of alcohol to one part of water) at 59° F. Cripps places special importance upon this last test, as it enables the recognition of adulteration with cedarwood oil or castor oil.

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* Pharm. Jour. and Trans., 1892, 461.

East Indian sandal-wood oil dissolves clear in 10 to 12 parts by weight of 70 per cent. alcohol at 68° F. West Indian sandal-wood oil, under similar conditions, only gives an opaque solution with 50 to 70 parts alcohol. Cedar-wood oil is not soluble even in 100 parts of 70 per cent. alcohol.

Sandal-wood oil is chiefly used in medicine, and also in perfumery.

### *Aristolochiaceæ.*

*Aristolochy oil* (*Osterluzeiöl*, G.) The root of *Aristolochia Clematitis*, L., was formerly officinal. From the root and herb of the plant a thickly-fluid, golden-yellow oil, having a specific gravity of 0.9 and an acid reaction, may be obtained.

*Snake-root oil, Virginian* (*Schlangenwurzelöl*) Virginia snake-root, *Aristolochia Serpentaria*, Jacq., consists of a rhizome which is about 1 inch long, ⅛ inch thick, is bent up and down and bears on the upper side the short, approximate stem-remnants which are flat or slightly convex above. It was used by the American aborigines as a cure for snake bites (whence its name), and by the early colonists as a tonic and stimulant. The root contains 0.5 to 2 per cent. of a pale brown volatile oil having at the same time a taste and odor of valerian and camphor, and a specific gravity of 0.988 at 59° F.

*Asarum oil* or *hazelwort oil, oleum asari radicis* (*Haselwurz- or Asarumöl*, G.; *essence d'asaret*, F.) The root of hazelwort, *Asarum europæum*, L. was formerly used in medicine. The plant is distributed throughout Europe and grows in shady places. The root has a peculiar, camphoraceous odor and a peppery, very pungent taste. By distillation the root yields about 1.08 per cent. of oil.

By standing, asarum oil separates large quantities of asarum-camphor. When treated with a little alcohol two layers are formed, one containing asaron and some volatile oil in solution, and the other consisting of volatile oil. By distilling the latter layer over slaked lime a distillate is obtained from which asaron crystallizes out. After separating the asaron, the actual volatile oil remains back as the liquid portion. It is somewhat viscous, of a yellowish color, sharp, pungent taste, and a valerian-like odor. It dissolves readily in alcohol and ether (Maier).

According to A. S. F. Petersen, asarum oil forms a thickly-fluid, somewhat turbid oil of a dark brown color, a sweetish, aromatic, not disagreeable odor, and entirely neutral reaction. At 1.4° F. the oil becomes stiff and salve-like without separating crystals; but by long standing at a low temperature a considerable quantity of asaron crystallizes out. The oil has a specific gravity of 1.046 and commences to boil at 338° F. By distilling the oil with steam about 16 per cent. of a light oil floating upon water passes over, which is succeeded by a small quantity of heavier oil, the principal mass remaining in the residue. By distilling asarum oil by itself considerable decomposition takes place at about 572° F., the products of which form green and blue oils; they become, however, decolorized by keeping.

Asarum oil consists of levo-pinene, $C_{10}H_{16}$ (only 4.5 per cent.), methyl eugenol, $C_6H_3C_8H_5(OCH_3)$, (perhaps also methyl iso-eugenol, principal constituent), and varying quantities of asaron, $C_6H_2C_3H_5(OCH_3)_8$ (at all events in smaller quantities).

Asarum oil dissolves iodine, with heating and disengagement of yellow red vapors, a very viscous, yellow-brown resin being finally formed. Nitric acid, on heating, colors the oil greenish yellow brown, then red brown, after which, with violent reaction, conversion into a red brown resin takes place. Sulphuric acid thickens the oil and colors it brownish red to blood red.

Asarum oil from the entire plant was obtained by Zeller, the entire flowering plant being subjected to distillation. The oil was liquid and, even when kept for fifteen years, never showed inclination towards crystallization. The oil was also free from asaron. The yield was only 0.2 per cent.

*Canadian asarum oil* or *Canadian snake-root oil* from ***Asarum canadense*** possesses, according to Power, a different composition from the European asarum oil. It contains little ***asarene***, $C_{10}H_{16}$; further *asarol*, $C_{10}H_{18}O$, which is probably a phenol; further, together with the asarol, ester of acetic acid, and probably also ester of valerianic acid. The yield from the root is 2.8 to 3.3 per cent. The oil has a specific gravity of 0.93 to 0.96 at 59° F.

## XI. *Volatile Oils, the Disposition of which in the Previously Discussed Groups was not Possible.*

*Mew oil* (*Bärwurzelöl*, G.) is the distillate from the root of *Meum athamanticum*, Jaqu. The yield is 0.67 per cent. The oil is dark yellow, possesses an odor reminding one of lovage oil, has a specific gravity of 0.999 at 69.8° F., and boils between 338° and 572° F.

*Damiana leaf oil* (*Damianablätteröl*, G.) The drug known in commerce as damiana leaves is said to be derived from *Turnera microphylla*, D. C., and other species of *Turneraceæ.* Schimmel & Co. obtained from the leaves 0.9 per cent. of a viscous, greenish oil possessing a chamomile-like odor, a specific gravity of 0.986 at 59° F., and boiling between 482° and 590° F. The higher boiling portions of the oil contain "blue oil." Damiana leaves are said to act powerfully upon the sexual organs; whether the volatile oil participates in this action is not known.

*Dilem leaf oil* (*Dilemblätteröl*, G.) According to Schimmel & Co., the dilem leaves brought from Java possess a very fine odor and by distillation yield 0.9 to 1 per cent. of a volatile oil which, as regards odor, resembles patchouly oil, but smells fresher, finer and less musty. It is yellowish green, quite thicky-fluid, has a specific gravity of 0.96 at 59° F., and boils between 482° and 572° F.

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*Erechthitis oil* (*Feuerkraut or Erechthitisöl*, G.) is derived from *Erechthitis hieracifolia*. The rectified oil is, according to F. Power, colorless, highly refractive, and boils at from 365° to 374° F. According to A. Todd, it has a specific gravity of 0.845 to 0.855 and begins to boil at 353.3° F.; up to 361.4° F. only 5 per cent. distil off. The oil is very slightly levo-rotatory or very slightly dextro-rotatory. By distillation in a current of steam it leaves behind a pale straw-colored resin. According to Beilstein and Wiegand, the oil consists almost entirely of terpenes. From the portion boiling below 347° F., a terpene, $C_{10}H_{16}$, boiling at 347° F. and having a specific gravity of 0.838 at 65.3° F., could be isolated. With hydrogen chloride this terpene forms a liquid hydrochloride. The greater portion of the oil passes over only at a temperature above 392° F. The fraction between 464° and 590° F. also contained a terpene. Finally it may be remarked that, according to Power, the oil contains sesquiterpene, $C_{15}H_{24}$.

*Musk seed oil* (*Moschuskörneröl*, G.) is obtained by distilling with steam the musk seed brought into commerce chiefly from Java and the Antilles. The yield is 0.1 to 0.25 per cent. The oil has a specific gravity of 0.900 to 0.905 at 77° F. and solidifies at the ordinary temperature. It has an odor of musk and appears to contain the same body which imparts the odor to musk. The oil contains very likely free palmitic acid. It cannot be distilled without suffering decomposition, and yields a very acid distillate, The oil freed from palmitic acid remains liquid at 32° F. Musk-seed oil is used in perfumery.

*Osmitopsis oil* (*Osmitopsisöl*, G.) is said to be derived from *Osmites Bellidiastrum* = *Bellidiastrum osmidoïdes* = *Osmitopsis asteriscoïdes*, probably a plant belonging to the *Compositæ*. The crude oil is limpid, feels rough between the fingers, and has a greenish-yellow color. Its odor is penetrating and reminds one of camphor and cajeput oil. Its taste is burning and acrid. It has a neutral reaction and does not separate stearoptene even when exposed to a low temperature. It has a specific gravity of 0.931 at 59° F., and boils between 348.8° and 402.8° F.

Between 402.8° and 406.4° F. a small quantity of camphor collected in the neck of the retort, while a resinous mass remained behind in the retort itself. The portion obtained by rectification between 352.4° and 359.6° F. possessed the properties and composition ($C_{10}H_{18}O$) of cajeputol (cineol). Osmitopsis oil is in every respect related to cajeput oil.

# CHAPTER XXI.

## TESTING VOLATILE OILS.

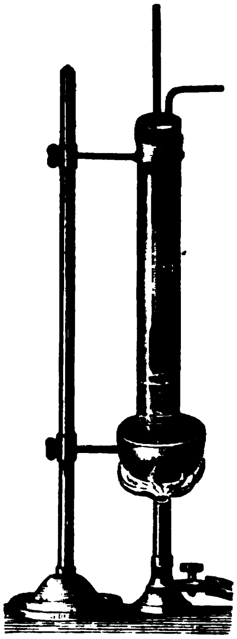
MANY volatile oils, especially those occurring only in very small quantities in the flowers, command a very high price, while others, occurring in larger quantities in plants whose cultivation is easy and possible in temperate climates, are comparatively cheap. It is of course to be expected that this should lead to the adulteration of the more expensive oils with cheaper ones, and in fact there is no other chemical product which is so extensively and shamelessly sophisticated as volatile oils. In the most favorable case expensive oils are adulterated with large quantities of cheaper oils, having a similar odor, or with other substances difficult to detect; but frequently a cheap oil is not only sold under the name of an expensive oil, which in itself is a fraud, but the cheap oil is even adulterated with still cheaper ones.

That the detection of adulterations is connected with many difficulties will be seen from the description of the general properties of volatile oils, as it frequently happens that they are identical as regards their chemical composition, turpentine oil having, for instance, the same composition as many expensive oils.

The physical properties of volatile oils, such as the density, boiling point, and solidifying point, do not furnish, as in most other bodies, a reliable guide. The densities vary between very wide limits, and the boiling point of the same oil frequently from 60 to 90 degrees, according to its derivation and age. This is readily explained by the fact that many volatile oils do not consist of a single combination, but generally of two in varying quantities.

Though the physical properties do not furnish a reliable guide, they may, nevertheless, be found useful in testing volatile oils, as they at least indicate the maximum and minimum limits of the specific gravity and boiling point which an unadulterated oil should have, and for that reason they have been given.

FIG. 300.



The specific gravity of volatile oils is determined by the same means and in the same manner as given for fixed oils (Vol. 1, p. 59).

The boiling point is determined by inserting a long thermometer into a glass tube (Fig. 300), secured by a suitable support and suspended free at a small distance over a metal saucer, which can be heated by a gas or spirit flame. A few cubic centimetres of the oil to be examined are introduced into the glass tube, and the thermometer is then inserted so that the bulb is suspended closely over the level of the fluid without, however, touching it. In the cork which holds the thermometer is a tube bent at a right angle and open on both ends.

To determine the boiling point the small saucer beneath the tube is heated until the development of gas bubbles indicates the beginning of the boiling of the oil. The flame is then so far moderated as to keep up the boiling, and the heating continued till no further change is observed in the thermometer. The degree is then noted, but the boiling is continued and the degree again noted in five minutes. The boiling point is most accurately determined by taking the average of three or four of such observations.

It will be observed that many thickly-fluid oils commence to become transparent at a certain degree of temperature, but again lose their transparency on cooling to a certain degree.

Solid oils will melt at a certain temperature and solidify again at a temperature frequently below the melting point. All such conditions must be taken into consideration, as they frequently furnish important guides in regard to the purity of the volatile oil.

*Solidifying point.*—Most volatile oils solidify on cooling to a certain temperature, either completely or only one portion of them, while another remains fluid even at a much lower temperature. The first portion, as previously stated, is called stearoptene and the second eleoptene. Many volatile oils solidify at a comparatively high temperature, for instance, the oils of aniseed, fennel and rose, while others do not solidify at all, or only partly, even at a low degree of temperature.

To determine the solidifying points of oils which become solid only at a low temperature, use is made of a freezing mixture prepared from pounded ice and common salt, calcium chloride, or ammonium nitrate. A thin glass vessel containing a tube with the oil to be examined and a spirit thermometer is placed in the cold mixture in a large vessel. The tube with the oil is occasionally taken out to see whether a separation of crystals or complete solidification takes place, and the temperature is noted when such is the case.

*Determination of water.*—Add to the oil twice the volume of benzene; in the presence of water turbidity caused by small drops of water takes place. Instead of benzene it is preferable to employ 3 to 5 times the volume of petroleum-ether of specific gravity 0.67 to 0.675. According to J. Kulinsky, add to 15 or 20 drops of oil a piece of tannin the size of a pea; in the presence of water the tannin deliquesces.

The most common adulterations of volatile oils, beside those with cheaper volatile oils, are fatty oils, resins, spermaceti, alcohol, chloroform, paraffine, or wax.

*Detection of fatty oils, resins, etc.*—This is the clumsiest of all adulterations and can be detected without much difficulty. The test is based upon the fact that nearly all volatile oils are soluble in strong spirit of wine, which is not the case with fatty oils, the only exception being castor oil, which is soluble in alcohol and is, therefore, most frequently used for the adulteration of volatile oils.

To detect the presence of a fatty oil, place a drop of the suspected sample upon white paper and expose it for a short time to heat. If it evaporates completely the oil is pure. But frequently a transparent stain remains with old oils without their being adulterated, which is due to resin formed by the absorption of oxygen and remaining dissolved in the oil. In this case a transparent ring is generally formed by the concentration of the resin on the edges of the stain.

If no tangible results are obtained by this test, pour a few cubic centimeters of the oil upon a watch-glass and heat it very slowly upon a piece of sheet iron until all the odor has disappeared. If the watch-glass becomes empty in a short time nothing but volatile oil was present, but if a viscous residue remains this may consist either of fatty oil or resin, or of both.

Treat the residue with strong alcohol; if it dissolves it may be resin or castor oil. Dilute the solution with much water; a white flocculent turbidity indicates resin; the separation of an oily liquid, after standing, castor oil. If the residue remains undissolved it consists of a fatty oil, generally oil of almond or olive.

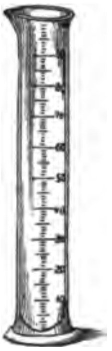
The presence of castor oil can be accurately determined by bringing the residue from the watch-glass into a test-tube by means of a glass rod and compounding it with a few drops of nitric acid. A heavy evolution of gas takes place, after the cessation of which solution of soda is added as long as there is any sign of effervescence. If the added fatty oil was castor oil the contents of the test-tube will show a peculiar odor due to œnanthylic acid formed by the action of nitric acid upon castor oil.

For the *quantitative determination of fatty oils*, Rhien gives the following method: Bring into a flask of 200 cubic centimeters capacity 100 ccm. of distilled water and 1 to 15 ccm. of volatile oil. Close the flask with a doubly-perforated stopper.

Through one of the perforations a tube, through which steam is introduced, passes to the bottom of the flask; from the other perforation a tube leads to a Liebig cooler. A small Florentine flask, with cylindrical vessel graduated in $\frac{1}{10}$ cubic centimeter, is placed before the lower end of the cooling pipe. The volatile oil, together with the water of condensation, collects in the receiver, and the quantity is read off in cubic centimeters. The difference between the cubic centimeters received and used is calculated to fatty oil. The operation requires 10 to 15 minutes. The presence of alcohol, however, may lead to inaccurate results.

*Detection of alcohol or spirit of wine.*—A practical test to quickly show the presence of large quantities of alcohol in volatile oils is as follows: Fill a cylinder graded into 100 cubic centimeters (Fig. 301) to the 10 mark with the oil to be examined and add water to the 50 mark. Shake thoroughly and allow the mixture to rest until the fluids separate. A decrease in the volume of oil proves the presence of alcohol, the latter having passed into the water. Mr. E. B. Stuart recommends the following test as having proved most useful for the detection of small quantities of alcohol. Take a test tube, fit a cork tightly, through which a glass tube bent at a right angle at its middle has been passed. The free end of the tube is placed in another test-tube. The corked test-tube contains the suspected oil, and is placed in a water-bath and a gentle heat applied. At a temperature of about 175° F. any alcohol present will distil over and may be condensed in the second test-tube, which is partially immersed in water of as low a temperature as can be conveniently obtained. If any alcohol distils over, it can be usually identified by its sensible properties, taste, smell, etc.; but if not, the liquid should be colored by the addition of a drop or more of compound tincture of iodine. Liquor potassa is then added drop by drop until the color disappears, the solution having been previously warmed to about

FIG. 301.



blood heat. On standing a yellow sediment of iodoform will subside if even traces of alcohol were present.

A. Drechsler recommends the use of a solution of 1 part of potassium bichromate in 10 parts of nitric acid of 1.30 specific gravity. Alcohol, if present, is at once betrayed by the pungent odor of ethyl nitrite.

*Detection of chloroform.*—Place the oil to be examined in a test-tube closed with a cork through which a glass tube bent at a right angle has been passed. In the open end of the glass tube place a moist strip of paper coated with paste in which potassium iodide has been dissolved. By heating the contents of the test-tube to about 158° F. the chloroform evaporates, [illegible] present in the oil, the originally white strip of [illegible] show a blue coloration.

[illegible] another test the oil is dissolved in alcohol and [illegible] with zinc and dilute sulphuric acid. After [illegible] water and separate the aqueous [illegible] from the oil by passing it through a wet filter. [illegible] is then tested for chlorine by adding nitrate of silver [illegible] acid.

*Detection of turpentine oil.*—The following method is based upon the readier solubility of oil of turpentine in alcohol than most other volatile oils. Shake about 10 grammes of the suspected oil with 40 grammes of [illegible] per cent. alcohol. After the liquid has again separated into two layers, pour the upper one, which is the alcoholic solution of the turpentine oil, into a porcelain saucer previously weighed and heated to about 158° F. The alcohol evaporates very rapidly, leaving the pure turpentine oil behind.

The adulterations of volatile oils with oil of turpentine are almost incredible; much so-called caraway oil of commerce being actually nothing but rectified oil of turpentine compounded with just sufficient genuine oil to give the mixture the characteristic odor.

Another method for the detection of turpentine oil is as follows: Place about 50 grammes of poppy-seed oil in a granulated glass tube, and add an equal quantity of the volatile oil to be examined. After shaking the mixture thoroughly allow it to stand. If the oil is pure the mixture becomes milky, and does not become clear until after a few days; but if oil of turpentine is present, even as little as 5 per cent., the mixture remains transparent.

Another method for the detection of oil of turpentine is H. Hager's guaiacum test. Oil of turpentine, or volatile oil containing it, when heated with finely-powdered guaiacum to nearly the boiling point, shows a dark violet color, caused by the action of ozone. Three classes of oils may be distinguished: namely, oils inclining towards the formation of ozone; oils which directly incite oil of turpentine to the formation of ozone; and oils which, with a content of oil of turpentine, are indifferent toward guaiacum. Hager has published an extensive treatise on this subject,* and has recently suggested some modifications which will be briefly mentioned: 1. Bring into a glass-tube 0.6 to 1 gramme of freshly powdered guaiacum (*Resina Guajaci nativa sive naturalis*), boil with 10 to 20 drops of ethyl alcohol, add 5 to 10 drops of amyl alcohol, 20 to 30 drops of volatile oil, and 1 cubic centimeter of benzol; repeat the test for comparison, but add 4 or 5 drops of oil of turpentine. Keep the samples excluded from daylight. 2. Ten drops of oil, 2 cubic centimeters of guaiacum tincture and 10 drops of amyl alcohol, and a similar sample for comparison with the addition of 2 drops of pure oil of turpentine. If the coloration is the same in both tubes, the oil is adulterated with oil of turpentine. Sunlight has to be excluded. *Guaiacum tincture:* 3 grammes of freshly-powdered guaiacum, 1.25 grammes freshly-powdered purified guaiacum (*Resina Guajaci alcohole depurata*), 120 cubic centimeters benzol. Solution has to be very gradually effected in the dark; do not shake, but simply move in a circular manner. Allow to settle and decant. Repeat the test with ordinary guaiacum tincture: oil 10 drops, guaiacum tincture 2 cubic centimeters, and 10 drops amyl alcohol; to the controlling sample add 2 drops of oil of turpentine. The presence of oil of turpentine can be safely assumed only when the blue coloration appears simultaneously. *Ordinary guaiacum tincture:* 4 grammes freshly-powdered guaiacum (*Resina Guajaci nativa sive naturalis*), and 120 cubic centimeters benzol, treated in the same manner as above. Under certain conditions, the reaction has to be promoted by the addition of 10 drops more of amyl alcohol, and also by heating to 122° F.

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* Chemische Reactionen zum Nachweis des Terpentinöles in den ätherischen Oelen, Berlin, 1885.

*Detection of paraffin, wax, and spermaceti.*—These substances not being volatile at the temperatures at which the volatile oils boil, can be detected by distillation, after which the presence of the particular adulterant is ascertained by the fusing and solidifying points; paraffin melts at 124° F., wax at 149° F., and spermaceti at 113° F.

### *Other Testing Methods.*

Many tests have been proposed by chemists for the purpose of determining whether a volatile oil is genuine or adulterated. Though these tests are very characteristic for many volatile oils, it will be readily understood that on account of the difference in the chemical constitution of the oils, they do not suffice for all.

*Test with nitro-prusside of copper.*—This test is chiefly used to distinguish oils free from oxygen from those containing oxygen. By boiling a small piece of nitro-prusside of copper with the oil to be examined, the fluid, with oil containing oxygen, acquires a dark coloration, and the precipitate formed is brown or variegated. The color of oil free from oxygen remains unchanged, and the precipitate has a beautiful green or blue color.

*Iodine test.*—Various phenomena are observed by bringing volatile oils together with iodine. The test is made by placing a grain of iodine as large as a pin-head, together with 5 or 6 drops of the oil, upon a watch-glass. Many oils detonate with iodine, considerable heat being developed; others slowly evolve vapors with but slight heating, and many have no effect upon it, but generally dissolve it more rapidly.

*Alcohol and sulphuric-acid test.*—By shaking volatile oil with about one-sixth of its volume of strong sulphuric acid, more or less heating takes place. After allowing the fluid to stand until it is thoroughly cooled, four to five times the quantity of strong alcohol is added, and the whole thoroughly mixed by shaking. The mixture is then either turbid or transparent, changed in color, and forms a body which is either soluble or insoluble in boiling spirit of wine.

*Sodium Test.*—Sodium metal placed in oils free from oxygen remains unchanged and does not alter the oils; from oils containing oxygen it evolves hydrogen gas, the evolution being very violent in the presence of alcohol, and the mixture becoming dark and viscous in a short time.

*Solubility in alcohol.*—Dr. Hager describes his method of testing volatile oils as to their purity by their solubility in alcohol as follows: Mix 1 volume of volatile oil at about 60° to 60.5° F. with 2 volumes of absolute alcohol (specific gravity 0.799). When the mixture has become clear add diluted alcohol of 0.889 specific gravity, containing 70.9 per cent. by volume of absolute alcohol, in small portions, or in drops, until the mixture has become, after one minute, so far turbid that it only appears opalescent, when agitated, without being milky. In many cases the further addition of a drop of diluted alcohol is sufficient to render the opalescing mixture milky-white. If the opalescence at the above temperature is accompanied by flocculent particles, in the case of oil of anise, rose, and similar oils, the adulterant may be spermaceti, paraffin, or other such bodies.

If the mixture is turbid, but still translucent, more of the diluted alcohol is added until the proper point is reached. It should be barely translucent.

The alcohol test permits the recognition of adulteration in most cases, though not always the precise adulterant. The latter must be sought for by other means.

The following list is given by Dr. Hager with the statement that the results are based upon tests made with two or three kinds of each oil. Where $x$ is quoted, the oil is completely soluble in the diluted alcohol. If the mixture of the oil with two volumes of absolute alcohol is turbid or milky, this is especially stated. The specific gravity of the oils examined is also given.

| A mixture of 1 volume of volatile oil and 2 volumes of absolute alcohol of 0.799 specific gravity. | Specific gravity. | Require to be rendered opalescent, volumes of dilute alcohol of 0.889 specific gravity. |
|---|---|---|
| Benzol (soluble in 9 volumes of the dilute alcohol). | | |
| Carbon disulphide | 1.272 | 0.8 to 0.9 |
| Chloroform | 1.495 | 10 “ $x$ |
| Nitrobenzol | 1.185 | 10 “ $x$ |
| Oil of almonds, bitter | 0.960 | 10 “ $x$ |
| “ amber, rectified | 0.858 | 0.3 “ 0.5 |
| “ angelica root | 0.898 | 0.5 “ 0.7 |
| “ “ seed | — | Milky white. |
| “ anise, Russian | 0.981 | 1.3 to 1.5 |
| “ “ very old | 0.990 | 10 “ $x$ |
| “ “ star, fresh | 0.976 | 0.8 “ 1.0 |
| “ bergamot | 0.875 | 1.0 “ 1.3 |
| “ cade | 1.005 | 0.05 “ 0.15 |
| “ cajeput | 0.920 | 3.0 “ 4.0 |
| “ “ green | 0.904 | 8.0 “ 10.0 |
| “ “ old | — | 5.0 “ 8.0 |
| “ calamus | 0.920 | 0.9 “ 1.1 |
| “ cardamon | 0.980 | 1.5 “ 2.0 |
| “ caraway | 0.945 | 3.0 “ 5.0 |
| “ “ old | 0.955 | 8.0 “ 10.0 |
| “ “ rectified | 0.903 | 1.8 “ 2.0 |
| “ cinnamon, cassia | 1.030 | 2.0 “ 2.5 |
| “ “ Ceylon | — | 15.0 “ $x$ |
| “ cloves | 1.060 | 10.0 “ $x$ |
| “ copaiva | 0.920 | 0.3 “ 0.35 |
| “ coriander | 0.880 | 5.0 “ 10.0 |
| “ cubeb | 0.945 | Turbid mixture. |
| “ “ | 0.920 | 0.05 to 0.1 |
| “ curled mint | 0.940 | 0.8 “ 1.1 |
| “ dill | 0.880 | 3.5 “ 5.0 |
| “ eucalyptus | 0.900 | Milky turbid. |
| “ fennel | 0.990 | 0.8 to 1.1 |
| “ “ very old | — | 1.3 “ 1.5 |
| “ juniper berries | 0.850 | Milky turbid. |
| “ “ wood | 0.860 | 0.5 to 0.75 |
| “ lavender | 0.890 | 2.0 “ 2.5 |
| “ “ old | 0.888 | 10.0 “ $x$ |

| A mixture of 1 volume of volatile oil and 2 volumes of absolute alcohol of 0.799 specific gravity. | Specific gravity. | Require to be rendered opalescent, volumes of dilute alcohol of 0.889 specific gravity. |
|---|---|---|
| Oil of lemon | 0.870 | 0.2 to 0.4 |
| " lemon-grass | 0.888 | 6.0 " 10.0 |
| " limes | 0.900 | 0.15 " 0.3 |
| " mace | 0.895 | 0.6 " 0.9 |
| " marjoram | 0.901 | 1.5 " 2.5 |
| " melissa | 0.878 | 3.0 " 3.3 |
| " mustard | — | 10.0 " *x* |
| " neroli | 0.870 | 2.5 " 3.3 |
| " orange, sweet | 0.850 | 0.3 " 0.5 |
| " " bitter | 0.876 | 0.35 " 0.5 |
| " parsley | 0.950 | 1.0 " 1.3 |
| " patchouli | 0.980 | 0.4 " 0.5 |
| " peppermint | 0.915 | 1.2 " 1.9 |
| " " very old | 0.925 | 5.0 " 6.5 |
| " rose | 0.860 | 0.4 " 1.2 |
| " rosemary, French | 0.894 | 2.5 " 2.8 |
| " " Italian | 0.904 | 4.0 " 5.0 |
| " rue | 0.890 | 4.0 " 5.0 |
| " sage | 0.920 | 1.5 " 1.8 |
| " sandal | 0.980 | 4.0 " 5.0 |
| " sassafras | 1.060 | 1.7 " 1.8 |
| " " very old | 1.080 | 3.5 " 4.0 |
| " savin | 0.898 | 0.5 " 0.7 |
| " tansy | 0.920 | 2.0 " 2.5 |
| " thyme | 0.895 | 1.0 " 1.4 |
| " turpentine | 0.890 | Milky turbid. |
| " valerian | 0.970 | 3.5 to 4.5 |
| " verbena | 0.895 | Milky turbid. |
| " vetiver | 0.923 | 0.9 to 1.1 |
| " wintergreen | 1.158 | 7.0 " 10.0 |
| " wormseed, levant | 0.920 | 10.0 " *x* |
| " wormwood | 0.965 | 3.5 " 5.0 |
| " ylang-ylang | 1.009 | 0.7 " 0.9 |

*Iodine pentabromide test.*—C. McC. Forney* applies this test by placing 5 or 6 drops of the volatile oil on a watch-crystal and adding 1 drop of the pentabromide. The latter is prepared by dissolving 127 grains of iodine in 400 grains of bromine. All the oils used were of the best commercial quality and pure. Corresponding experiments were performed also with the same volatile oils previously mixed with 25 per cent. oil of turpentine and the same proportion of 95 per cent. alcohol. The following table shows the results:

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* American Journal of Pharmacy, November, 1882.

| OILS. | PURE OILS. | | OILS + 25 PER CENT. OIL OF TURPENTINE. | | OILS + 25 PER CENT. ALCOHOL. | |
|---|---|---|---|---|---|---|
| | Reaction. | Color. | Reaction. | Color. | Reaction. | Color. |
| Almond, bitter.... | None | Orange-red. | Slight reaction and sputtering | Brownish-yellow. | None | Orange-red. |
| Amber, rectified .. | Slight | Cloudy green; dark precipitate. | Brisk; effervescence; slight sputtering | Brown-red. | Slight effervescence | Cloudy olive; black precipitate. |
| Anise ............ | Violent; sputtering | Brown-red. | Very violent | Yellowish-brown. | Brisk, slight effervescence and sputtering | Brown-red. |
| Bay ............ | Violent; brisk effervescence; sputtering | Brownish-green. | " " | Colorless or yellowish. | " " " | Olive-green. |
| Bergamot ........ | " " | Olive-green. | " " | Brownish, then greenish-yellow. | " " " | Brownish. |
| Camphor ......... | " " | Brownish-red. | " " | Brownish-red. | Brisk effervescence | Brown-red. |
| Caraway .... .... | " " | Greenish-yellow. | " " | Reddish-brown. | Slight effervescence | Cloudy, brownish-yellow. |
| Cassia............ | Slow, slight effervescence | Greenish-brown. | Brisk effervescence | Cloudy, blackish-brown. | " " | Yellowish to greenish-brown |
| Cinnamon, Ceylon. | Violent; brisk effervescence; sputtering | Cloudy, blackish-brown. | Very violent | Olive-green, then greenish-black. | " " | Brown-red. |
| Cloves ........... | " " | Brown sediment. | " " | Dark brown. | Brisk; slight effervescence | Cloudy, yellow. |
| Copaiba .......... | Slight; gentle effervescence; green vapors | Green. | Brisk effervescence; green vapors | Brownish, then olive-green, black precipitate. | Slight; green vapors | Green. |
| Croton ........... | None | Greenish-yellow. | Brisk effervescence | Brown-red. | None | Cloudy, yellow. |
| Cubeb............ | Violent; brisk effervescence; sputtering | Greenish-yellow, dark green upon mixing or stirring. | Very violent | Cloudy, inky, then clear, olive-green. | Like pure oil | Dark to light green. |
| Fennel ........... | " " | Brown-red. | " " | Dark brown. | Slight effervescence | Red-brown. |
| Hemlock ......... | " " | Brownish-yellow. | " " | Brownish-yellow. | Brisk; slight effervescence and sputtering | Cloudy, yellowish-brown. |
| Horsemint........ | " " | Brownish-red. | " " | Straw-yellow. | Brisk effervescence | Brownish-red |

| OILS. | PURE OILS. | | OILS + 25 PER CENT. OIL OF TURPENTINE. | | OILS + 25 PER CENT. ALCOHOL. | |
|---|---|---|---|---|---|---|
| | Reaction. | Color. | Reaction. | Color. | Reaction. | Color. |
| Juniper .......... | Violent; brisk effervescence; sputtering | Green; black precipitate. | Very violent | Sea-green. | Brisk; slight effervescence and sputtering | Green and brown layers; after mixing, dark green. |
| Lavender......... | " " | Greenish-brown. | " " | Greenish-brown. | Brisk; slight effervescence | Yellowish-brown. |
| Lemon........... | " " | Brownish-yellow. | " " | Cloudy, dark yellow. | Brisk; slight effervescence and sputtering | Yellow and brown layers; not miscible. |
| Orange........... | " " | Black precipitate. | " " | Cloudy, dirty violet; then clear, dark yellow. | " " " | Brownish-yellow; brown precipitate. |
| Pennyroyal ...... | " " | Cloudy, blackish-brown. | " " | Dark brown. | Like pure oil; less sputtering | Dark brown. |
| Peppermint....... | " " | Cloudy, blackish-brown. | " " | Cloudy, purplish-black; then blue-black and inky green. | Brisk; slight effervescence and sputtering | Bright green, then cloudy. |
| Pimenta ......... | " " | Greenish brown. | " " | Greenish-brown. | " " " | Cloudy, greenish-brown. |
| Rosemary ........ | " " | Reddish-brown. | " " | Reddish-brown. | Like pure oil | Reddish brown. |
| Sassafras ........ | " " | Yellowish, then light gray. | " " | Reddish-yellow. | " " | Light brown. |
| Savin ............ | " " | Greenish-brown. | " " | Greenish-brown. | Brisk; slight effervescence and sputtering | Cloudy, yellowish-brown. |
| Spearmint ....... | " " | Brownish-yellow. | " " | Reddish-brown. | " " " | Brownish-yellow. |
| Spruce........... | " " | Dark brown. | " " | Reddish-brown. | " " " | Light brown. |
| Tansy............ | " " | Brownish-yellow, then colorless. | " " | Reddish-yellow. | " " " | Yellow and red layers; not miscible. |
| Wintergreen ..... | Slight; gentle effervescence | Reddish-brown. | More active than with pure oil | Straw color. | Slight effervescence | Cloudy, yellow. |

### *Color Reaction of Volatile Oils.**

I. SOLUTION OF BROMINE IN CHLOROFORM (1 in 20), in the proportion of 1 to 15 drops to 1 of oil, gives

*Colorless mixtures* with oils of turpentine, caraway, lemon, coriander, and cardamoms;

*Yellow mixtures* with bergamot, bitter orange, and neroli;

*Slowly turning green* with cloves, ginger, lavender, cajeput, cascarilla;

*Slowly turning greenish-blue* with oils of juniper, pepper, and galangal;

*Greenish-brown or brown mixtures* with oils of sweet marjoram, dill, cummin, and valerian;

*More or less fine rose, red, or reddish-violet tint* is gradually produced by oils of rosemary, fennel, anise, star anise, cinnamon, nutmeg, thyme, peppermint, myrrh, and parsley;

*Brownish-violet* with oil of mace;

*Blue or bluish-violet* with oil of cubebs, copaiba, laurel, sandal-wood, and sweet flag;

*Orange* with oil of wormseed, oil of cedar wood, and with camphor.

II. IMPURE CHLORAL HYDRATE (2 drops to 1 of oil) resembles the foregoing reagent in the coloration it produces with many oils. It differs, however, in its behavior to oils of lemon and bergamot, with which it assumes a reddish color; cloves, which turns red on warming; mace (fine rose-red); pepper (reddish-violet); copaiba (dark green); valerian (greenish); cummin (fine green); cinnamon (green with violet margin), and myrrh (reddish-violet).

III. ALCOHOLIC HYDROCHLORIC ACID varies in its action with the amount of acid it contains. A dilute solution is to be preferred, as the colorations, though appearing more slowly, are purer. Dilute alcoholic hydrochloric acid, in the proportion of 15 to 20 drops to 1 of oil, yields

*Colorless mixtures* with oils of turpentine, caraway, coriander, cardamoms, cloves, rosemary;

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* Dragendorff. "Plant Analysis," translated by Greenish.

*Yellow mixtures* with oils of bergamot (concentrated acid, orange to olive-green), mace (concentrated acid, reddish-brown), dill (concentrated acid, cherry-red), bitter orange, cummin (concentrated acid, deep violet);

*Brownish-red mixtures* with oils of cascarilla, lavender, sweet marjoram, wormseed, juniper (concentrated acid, red);

*Rose to deep red or reddish-violet mixtures* with oils of cubebs, pepper, copaiba, cedar wood, cinnamon, nutmeg, thyme, laurel, sweet flag, and myrrh;

*Red mixtures, turning blue,* with oil of peppermint.

IV. CONCENTRATED SULPHURIC ACID (2 or 3 drops to 1 of oil) assumes with most oils a yellow color, turning brown, and frequently passing finally to a fine red. The latter coloration is observable with oils of caraway, mentha crispa, sweet marjoram, star anise, mace, dill, juniper, cubebs, copaiba, sage, wintergreen, lavender, cascarilla, nutmeg, thyme, sandal-wood, peppermint, myrrh, and parsley;

*Violet color* is produced with oils of cardamoms, cloves, fennel, anise, cajeput, and laurel;

*Green and blue color* with oil of cinnamon.

V. FRÖHDE'S REAGENT (1 cubic centimetre of sulphuric acid with 0.01 gramme of molybdate of soda) resembles sulphuric acid in its action, but the colors are purer and make their appearance more rapidly. Very characteristic colors are produced with some oils by sulphuric acid mixed with one-sixth of its volume of a 5 per cent. aqueous solution of ferric chloride.

The oils should be dissolved in chloroform, in which the coloring matter is also soluble and thus seen to advantage.

*No coloration* in the chloroform, even after some lapse of time, is caused by oils of pennyroyal, parsley, coriander, fennel, anise, savin, and turpentine;

*Red tinge* with oils of ledum and peppermint;

*Violet or bluish-violet tinge* with ledum-camphor and oils of thyme, cajeput, galangal, pepper, cubebs, copaiba, and juniper;

*Green or bluish-green tinge* with oils of sweet marjoram, rosemary, caraway, dill, nutmeg, cloves, wormseed, and cinnamon;

*Olive-green tinge* with oil of bergamot, etc.

VI. FUMING NITRIC ACID gives especially characteristic colorations with oils of mace and nutmeg (blood-red), cubebs (green), copaiba (bluish-violet), gaultheria (cherry-red), cinnamon (carmine), myrrh (reddish-violet), pimento (blood-red), and pennyroyal (violet).

VII. PICRIC ACID (0.05 gramme to 5 to 6 drops of oil) is easily dissolved by some oils in the cold (caraway, cardamoms, cloves, rosemary, mentha crispa, sweet marjoram, anise, star anise, dill, valerian, cummin, gaultheria, cinnamon, sweet flag); by others only on warming. Some of the solutions deposit crystals on standing (turpentine, lemon, bergamot, sweet marjoram, mace, dill, galangal, bitter orange, wormseed, valerian, cedar wood, lavender, cajeput, nutmeg, thyme, laurel, and sandal wood); others gradually assume characteristic colorations:

*Olive-green*, oil of mentha crispa;

*Orange*, oils of cloves, sweet marjoram, anise, star anise, nutmeg, cinnamon, cummin, and thyme;

*Blood-red*, oils of fennel and myrrh;

*Brown*, oils of dill, cascarilla, and galangal;

*Reddish brown*, oil of wormseed;

*Deep brown*, oil of sweet flag;

*Deep grass-green*, oil of peppermint.

*Cohesion figures.*—Miss Kate Crane* has made experiments with the cohesion figures of volatile oils, and believes that they may be usefully employed as tests of the identity and purity of the oils. She is convinced that a little patient practice will teach the eye in a short time to detect the characteristic differences of the figures. To make these perfect it is necessary to observe the time in forming, for at *different periods* some varieties form figures very like, but with this precaution each is entirely characteristic. It is essential that the dish used, etc., be perfectly clean, so that when filled with water no dust or lint floats upon the surface, as this materially interferes with the perfect formation of the figure.

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* American Journal of Pharmacy, September, 1874.

A single drop is let fall from a burette, or glass rod, held steadily about four inches above the water upon the centre of the surface.

The experiments were made with a number of volatile oils, by themselves and mixed in different proportions with *oil of turpentine*.

The last-named oil, by itself, spreads instantly to the whole size of the plate, a common soup plate, and almost immediately the edge begins to break into irregular shapes, when a rapid motion takes place over the whole surface of the film; there seems to be a contest between the cohesion of the oil particles and the adhesion between them and the water. The oil makes repeated efforts to gather itself closer together, when the water instantly reacts, giving a wavy appearance to the whole figure. The play of colors at this point is beautiful, and serves to bring out the lines more perfectly. In a few seconds innumerable little holes appear over the surface, which soon are separated only by threaded lines, and the figure is like the most exquisitely fine lace.

*Oil of cinnamon* forms a figure not more than half the size of the last named. In a few seconds small portions are detached, and shortly separate into distinct drops, four or five larger and a number of smaller ones scattered about. With mixtures in different proportions of *oil of turpentine* the figures formed differently, taking more the characteristics of the adulterant as it predominated.

*Oil of nutmeg* forms a large figure instantly, the edge showing a beaded line. It gathers itself together and spreads again, very like oil of turpentine, but the surface presents more the appearance of watered silk. Within sixty seconds some holes appear, and in eighty more the surface is covered with them; these scarcely spread to more than a sixteenth of an inch in diameter, but from the first each is bordered with a dotted edge. The figure lasts some time without changing materially, except the openings lengthen out into an oblong shape, remaining entirely distinct. The play of colors is very fine.

With the addition of one-third the volume of *oil of turpentine* the first spreading is little different, but openings appear in half the time and the dotted border does not come so soon; in about four minutes the figure is most characteristically marked and soon breaks up entirely, this being the distinctive difference between the pure oil and the mixture.

*Oil of peppermint* spreads instantly to a large figure, and in ten or fifteen seconds openings appear, which increase rapidly in size; at first they look somewhat like the last named, but are not nearly so numerous, and the border soon is more like tiny drops. In one and a half or two minutes they begin to run together and the figure breaks up. With the addition of *turpentine oil* the figure forms more slowly, and the breaking up is less rapid, but in five minutes the outlines only remain.

*Oil of bergamot* spreads instantly, in thirty seconds tiny openings appear, not very abundant, and increase in size slowly; in five minutes they are not larger than oil of nutmeg at one and a half minutes. At first they have a dotted border, but as they increase in size this changes to a scalloped film, which spreads, until in eight or ten minutes they are joined together over the whole surface.

This with turpentine oil gives a watered surface in spreading, much more marked and with a finer play of colors.

*Hübl's iodine absorption process* (see Vol. 1, p. 201).—Mr. C. Barenthin* has applied Hübl's iodine methods for fixed oils to the examination of essential oils. He uses the following solutions:

1. Fifty grammes iodine and 60 grammes mercuric chloride in a liter of alcohol freed from fusel oil and let stand twelve hours.

2. Twenty-four grammes of hyposulphite of sodium in a liter of water.

3. A ten per cent. solution of iodide of potassium. Dissolve 0.1 to 0.2 gramme of the volatile oil in 10 cubic centimetres of chloroform, and add first 15 cubic centimetres of the iodine-

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* Archiv der Pharmacie, 1886, p. 848.

mercuric chloride solution; let stand three or four hours, and, in case the mixture gets discolored, a few more centimetres of solution are added. Now add 10 to 15 cubic centimetres iodide of potassium solution, dilute with 150 cubic centimetres of water, and titrate with hyposulphite till the mixture remains clear for about a minute. The iodide of potassium solution must be added before the water, and the relative proportions between this solution and the iodine-mercuric chloride solution must be 15 to 25 cubic centimetres.

According to the saturation of their constituents the volatile oils absorb different quantities of iodine, so that conclusions regarding the kind of oil under examination, or at least as to the presence of adulterations, can be drawn. According to R. H. Davies, the following oils absorb little or no iodine: Oils of almond, cassia, chamomile, cinnamon, wild thyme, pepper, mustard, American and Japanese peppermint, and wintergreen. English dill oil and caraway oil show at first a slow, but later on, more vigorous reaction. Immediate but slight reaction is exhibited by the oils of calamus, eucalyptus, pine needle, pennyroyal, sassafras, sandal-wood and spear mint. Vigorous reaction show the oils of anise, star anise, bergamot, copaiva, cubebs, fennel, juniper, lavender, lemon, marjoram, rosemary, rue, nutmeg, turpentine.

However, the iodine-absorption method has also its defects. As shown by R. A. Cripps, the numbers found by various authors vary considerably, this being due to the difference in the length of the time of action and also to the temperature. R. Williams recommends a conventional time of four hours for the oil to act upon the iodine solution; at the end of that period absorption, of course, is not finished, but in many cases is at least sufficient for the recognition of adulterations. Even a period of 48 hours would by no means assure the end of the reaction. In the appended table the iodine numbers found by some authors are given.

| Names of Volatile Oils. | Iodine number according to | | Derivation of the Oil. | Iodine number according to Davies. | Derivation of the oil. | Iodine number according to Williams. |
|---|---|---|---|---|---|---|
| | Barenthin. | Snow. | | | | |
| Anethol | — | — | from aniseed oil | 177.4–182.9 | — | — |
| " | | | from star anise seed oil | 177.8 | — | — |
| Aniseed | 164 | 121 | English | 189.7 | English | 185.9–274.4 |
| Bergamot | 260 | 345 | Commercial, 1888 | 276.1 | English | 247.9–283.7 |
| Bitter almond | — | — | English | 0 | — | — |
| Cajeput | — | — | — | — | Mitcham | 70.9–151.0 |
| Calamus | 155 | — | Commercial | 181.4 | — | — |
| Camphor | — | — | Commercial | 0.46 | — | — |
| Caraway | 265 | 233 | ? 1877 | 81.6 | Mitcham | 258.3–263.2 |
| Cardamoms | — | — | English | 139.3 | — | — |
| Cassia | — | — | Commercial | 159.5 | Mitcham | 70.7–75.1 |
| Cedar wood | — | — | — | — | Mitcham | 74.8–78.5 |
| Celery | — | — | English | 311.6 | — | — |
| Chamomile | — | — | English | 68.1 72.1 | — | — |
| Cinnamon | 100 | — | Commercial | 189.5 | Mitcham | 105.1–128.4 |
| Citronella | — | — | — | — | Mitcham | 186.3–191.3 |
| Clove | 270 | 467 | English | 349.5–362.5 | Mitcham | 155.46 |
| " | — | — | from stalks | 366.6 | " | 179.83 |
| Cubebs | — | — | English | 223.1–226.8 | — | — |
| Dill | — | — | English | 257.1 | — | — |
| Eucalyptus | 235 | — | — | — | Mitcham, Hitchin | 61.7–110.7 |
| Fennel | 140 | — | ? 1887 | 158.3 | — | — |
| Juniper | 245 | — | English, 1889 | 337.3 | Mitcham | 236.1–273.6 |
| " | | | Foreign | 363.9 | Hitchin | 250.06 |
| Lavender | 170 | English, 286 | English | 265.5–273.9 | Mitcham | 230.4–248.6 |
| " | | | French I$^{a}$ | 262.7 | Hitchin | 232.92 |
| " | | | French | 294.5 | French | 198.7–237.2 |

| Names of Volatile Oils. | Iodine number according to Barenthin. | Iodine number according to Snow. | Derivation of the Oil. | Iodine number according to Davies. | Derivation of the Oil. | Iodine number according to Williams. |
|---|---|---|---|---|---|---|
| Lemon | 285 | — | Prima | 328.3 | English, Mitcham | 320.04 |
| " | — | — | Six different kinds | 340.3–355.1 | English, Mitcham, Hitchin | 353.9–359.7 |
| Lemon-grass | — | — | — | — | English | 223.2–271.5 |
| Lime | — | 343 | — | — | English | 320.2–323 |
| Mace | 215 | — | — | — | English | 220.9-224 |
| Menthol | — | — | — | 0.12 | — | — |
| Nutmeg | — | — | English, 1889 | 308.3 | English | 201.9–206.2 |
| Orange | — | 362 | — | — | English | 341.8–347.7 |
| Origanum | 227 | — | — | — | — | — |
| Parsley | — | — | English, 1889 | 255.0 | — | — |
| Pennyroyal | — | — | Commercial | 188.9 | English | 78.6–85.9 |
| Peppermint | — | 64–179 | English, 1887–88 | 49.6–57.7 | — | — |
| " | — | — | Mitcham, 1888 | 81.9 | Mitcham | 36.8–46.4 |
| " | — | — | American | 121.8–143.9 | American | 53.1–71.7 |
| " | — | — | Japanese | 43.5–48.1 | Japanese | 37.1–56.1 |
| Rosemary | 185 | — | Commercial | 325.0 | English | 142.4–161.7 |
| Rue | — | — | — | — | — | 121.3–192 |
| Sage | 105 | — | — | — | English | 49.7–117.1 |
| Sandal-wood | — | — | English, 1888 | 226.6–233.9 | — | — |
| Sassafras | — | — | — | — | English | 151–162.7 |
| Savin | — | — | Commercial | 279.5 | — | — |
| Spike lavender | — | — | — | — | English | 207–287.9 |
| Thyme | 170 | — | — | — | Red | 168.4–181.5 |
| Thymol | — | — | Commercial | 171.5 | — | — |
| Turpentine | 300 | 397 | Commercial | 377.0 | — | — |
| Valerian | 80 | — | — | — | — | — |
| Verbena | — | — | — | — | English | 247.8–267.8 |
| Wild thyme | — | — | English | 254.9 | — | — |

*Iodol test.* According to Hirschsohn, weighed quantities of iodol are brought into a test-tube graduated in $\frac{1}{10}$ cubic centimeters, and small quantities of volatile oil gradually added until a clear solution results. The quantity of volatile oil required for the solution of 1 gramme iodol is then calculated. In making the experiments a temperature of 59° F. is maintained. Iodol is tetraiodopyrrol, $C_4H_5NI_4$. The oils of the Compositæ show the greatest solvent power, though it may be remarked that the solvent power of the oils is preponderatingly dependent on certain constituents of the oil. Thus for the solution of 1 gramme iodol 16 cubic centimeters of aniseed oil are required, but only 8 cubic centimeters of aniseed oil deprived of anethol. The results of some experiments are given in the table p. 632.

*Bromine addition.* This method has recently been recommended by A. Levallois. By pouring aqueous or alcoholic solution of bromine into water or alcohol containing a small quantity of volatile oil, the bromine solution is immediately decolorized until, with a continued addition of bromine, a point is reached at which the mixture acquires a yellow color, and the odor of volatile oil disappears. With an aqueous solution a white insoluble resin which, after stirring, remains adhering to the sides of the vessel, is at the same time separated. The quantity of bromine consumed is proportional to the quantity of dissolved volatile oil; but a correction has to be made, since for the perceptible coloration of 20 to 30 cubic centimeters of solution an excess of bromine of 0.2 to 0.3 ccm. of bromine solution is required. This method is especially suitable for the determination of the content of the waters from the distillation of volatile oils. About 1 quart of such water is distilled from a 2-quart balloon, through the cork of which is passed a pipe about 5 feet long and 7¾ inches in diameter. The pipe is inclined towards the flask and its end connected with a descending pipe. The first 20 or 30 cubic centimeters of distillate running off from the latter contain all the volatile oil. They are mixed with standardized bromine solution until a slightly yellow coloration appears; 0.2 to 0.3 cubic centimeter of bromine solution is then deducted and the rest calculated to bromine, whereby the content of the water is formed. The bromine solution is standardized with the same kind of volatile oil, the water of distillation of which is to be examined.

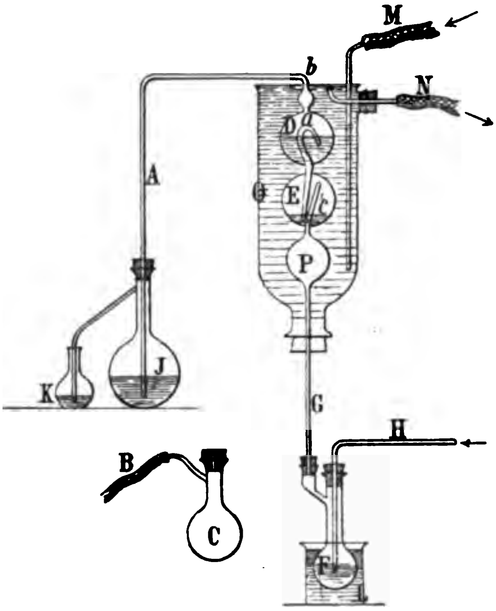
*Test with lyes.*—The volatile oils may occasionally be successfully tested by the saponification-method, *i. e.*, by determining the quantity of alkali which is neutralized by the oil. However, the point in question is not always an actual saponification (conversion of an ester into an alkaline salt of the acid and alcohol), but frequently only the neutralization of a free acid, formation of an alkaline phenol, etc. The term saponification number is applied to the number of milligrammes of potash lye consumed per 1 gramme of oil. R. Williams has boiled the oils with an excess of semi-normal alcoholic potash lye and titrated back the excess of alkali with semi-normal acid. The values found he has expressed in per cent. of oil, as shown in the table, p. 632. Generally speaking, the consumption of potash rises with the degree of oxidation. The most acid was bergamot oil (which contained citric acid); great variations, according to age, showed, for instance, sage oil, only old oil being rich in salviol and camphor. According to Kremel, the saponification number, for instance, for geranium oil is over 40.5, while for rose oil it is 0. According to E. Dieterich, the saponification number of volatile oils is of no value, since the limit between acid and ester number is insufficiently marked, and the figures found are throughout too low. Besides, fresh oil behaves differently from old oil.

*Maumené's test.*—This test (see Vol. 1, p. 181) is actually intended for fat oil, but R. Williams has successfully employed it also for volatile oils. Add 1.2 cubic centimeters of strong sulphuric acid to 6 cubic centimeters of volatile oil, and mix by stirring with a thermometer. Note the increase in the temperature. In some cases heating is so violent as to necessitate working with half the quantity of the mixture to prevent exhalation. If the volatile oils contain resin or drying oil, the increase in temperature is very marked. Compare column 4 of the table p. 632.

*Methyl number.*—R. Benedikt and A. Grüssner have recommended a method for the determination of the methyl number of volatile oils, which is based upon Seisel's method for determining the methoxylin organic combinations. A series of volatile oils contain methyl and ethyl esters, and hence, by boiling with hydriodic acid, yield alcoholic iodides. The ***methyl number*** indicates the number of milligrammes of methyl split off from 1 gramme of substance by boiling with hydriodic acid, whereby, to be sure, ethyl, propyl, etc., are also expressed in the equivalent quantity of methyl. 0.2963 gramme of clove oil yielded, for instance, 0.3737 gramme of silver iodide. Now 1 molecule silver iodide corresponds also to 1 molecule methyl, and hence, we have the proportion $AgI:0.3737 = CA_3:X$: for $AgI = 235$ and $CH_3 = 15$ results $X = 0.02385$ gramme. This value has to be calculated for 1 gramme of clove oil, hence, $0.2963 : 1 = 0.02385 : y$; from which results $y = 0.0805$ gramme. Finally the latter number has to be expressed in milligrammes, so that the methyl number of clove oil found is 80.5.

The apparatus used for the execution of the process is shown in Fig. 302. By means of its perforated stopper the flask *C* is first pushed over the pipe *A*. The flask *C* holds about 150 cubic centimeters and is charged with 0.5 gramme of finely triturated red phosphorus and some water, By blowing at *B*, this content of *C* is forced into the uppermost sphere *D*; from here, as soon as the small siphon *a* is filled, the liquid runs spontaneously into *E*. *D* and *E* hold 80 to 100 cubic centimeters. Rinse *C* in the same manner with distilled water until *E* is filled half full, and wash, by alternately blowing in and sucking of water, the pipe *A* until it is perfectly clean up to the bend at *b*. In the flask *F*, which has a capacity of 30 to 35 cubic centimeters, weigh off 0.2 to 0.3 gramme of volatile oil, and add a mixture of 10 cubic centimeters hydriodic acid of specific gravity 1.7, and 8 per cent. of acetic anhydride. Secure the flask *F* to the pipe *G*, which is 9 to 10 inches long and about 0.31 inch in diameter, conduct from *H* a slow current of carbonic acid through the entire apparatus, and place the flasks *J* and *K* before *A*, as shown in the illustration. The flask *J* holds about 120 cubic centimetres, and is charged with 5 cubic centimetres of a 40 per cent. nitrate of silver solution to which have been added 50 cubic centimetres of 95 per cent. alcohol free from fusel oil. *K* is charged with 1 cubic centimetre nitrate of silver solution and 10 cubic centimetres alcohol. The glycerin bath *L* is now heated, and water allowed to flow from *M* to *N* through the cooler *O*. The vapors from *F* ascend through *G* first into the bulb *P*, which has a capacity of about 50 cubic centimetres. In the bulb *P* the hydriodic acid and steam condense almost completely, and flow back through *G* into *F*. In *P* iodine is deposited in case it is liberated in larger quantities; a remnant of it passes together with methyl iodide

FIG. 302.



(ethyl iodide, propyl iodide, etc.) through *c* into *E*. By the pressure of steam the liquid in *E* is mostly forced into *D*, where the vapors must again pass the liquid. The phosphorus retains all the iodine, and pure methyl iodide reaches *J*, where it is absorbed with avidity by the nitrate of silver solution and yields a white precipitate of silver iodide and silver nitrate. When after one or two hours the liquid standing over the precipitate in *J* has become perfectly clear, the reaction is finished. *J* and *K* are first removed, and then *F*, and with the assistance of *C* the pipe *A* up to *b* is rinsed in the manner already described.

The contents of *C* are rinsed into a beaker of 200 cubic centimetres capacity, the alcoholic fluid from *J* and *K* is poured into a glass of 500 cubic centimetres capacity, the precipitate is washed twice or three times with 30 cubic centimetres of water each time, and, after the yellow silver iodide has settled, decanted into the first smaller beaker. The contents of the latter are mixed with 20 cubic centimetres of dilute nitric acid (½ acid, ½ water) and heated, while the contents of the larger beaker are diluted with water to 300 cubic centimetres, mixed with a few drops of nitric acid, and freed from alcohol by heating; they are then again brought up to 300 cubic centimetres by the addition of water. The precipitate of silver iodide from the two beakers together with that originally present is then collected upon a tared filter which is dried at 248° F., and again weighed.

Commercial hydriodic acid previously to being used for the experiments has to be boiled for two hours on the reflux cooler, which is fed with water of 158° F., otherwise the hydriodic acid frequently yields precipitate of silver iodide by itself. The temperature in *O* is best maintained at 158° F.; for this purpose the water entering at *M* is allowed to flow first through a heating flask. The use of warmed cooling water is conditional on the boiling point of the alcoholic iodides: methyl iodide, 108.5° F.; ethyl iodide, 160.3° F.; propyl iodide, 215.6° F.; iso-propyl iodide, 192.2° F.

Of course, only oils free from alcohol should be used for examination; when, however, the methyl number of the pure oil is known, the content of alcohol in an oil containing it can be quantitatively determined by the methyl number of such oil. The methyl number of absolute alcohol is 326.

Sulphuretted oils yield in the silver salt solution a heavy black precipitate, so that the mere weighing of the latter furnishes no reliable results. Oils with esters of higher alcohols do not give a clear liquid in *J*, even after heating for several hours.

*From the methyl number may be directly calculated:* 1. *The content of eugenol* in the oils of clove, cinnamon leaf, and Ceylon cinnamon; 2. *The content of anethol* in the oils of anise, star anise and fennel; 3. *The content of apiol* in parsley oil, when the methyl number of pure apiol has been determined. Eugenol, namely, contains a methoxyl: $C_6H_3(C_3H_5)(OCH_3)OH$, and anethol, also a methoxyl: $C_6H_4(C_3H_5)(OCH_3)$, while apiol appears to contain two methoxyls, probably: $C_6HC_3H_5(OCH_3)_2O_2CH_2$.

Fifty-seven determinations made by Benedikt and Grüssner led to the following results:

1. *Oils whose methyl number is* = 0: Oils of angelica, bergamot, bitter almonds, Hänsel's patent lemon, eucalyptus, geranium, rectified cherry laurel, copaiva, coriander, crisped mint, cubebs, Hänsel's patent-caraway, dwarf pine, best lavender, orange flower, peppermint, pennyroyal, wild thyme, rosemary, savin, sage, sandal-wood, turpentine, juniper, valerian, wormwood.

2. *Oils whose methyl number is 1 to 30:* Hänsel's patent lavender (2.4), Hänsel's bergamot (6.6), Schimmel & Co.'s extra strong caraway (6.6), cassia flower (6.7), orange peel (6.9), caraway chaff (8.3), olibanum (9.1), elemi (12.1), cherry laurel oil free from prussic acid (13.8), birch tar oil (22.4), Metzner & Co.'s prime lemon (23.6), calamus (24.2), Ceylon cinnamon (25.7).

3. *Oils whose methyl number is 60 to 100:* Hänsel's patent fennel (65.7), cassia leaf (73.2), clove stalk (73.8), star anise (79.8), aniseed oil (82.8), Hänsel's patent clove oil (86.9),

Schimmel & Co.'s technical eugenol (88.8), artificial wintergreen (89.4), parsley (92.2). See also appended table.

| Names of volatile oils. | *Iodol test.* | *Alkali test.* | *Maumené's test.* | *Methyl number.* |
|---|---|---|---|---|
| | 1 gramme iodol dissolves in cubic centimeters of volatile oil. | 100 grammes of oil require grammes of caustic potash. | Heating of the oil ° C. by $H_2SO_4$. | Milligrammes methyl in 1 gramme of oil. |
| Anethol | 16 | — | — | — |
| Anise | 16–17 | 0.25–0.38 | 68.69 | 82.8 |
| Bay | 20 | — | — | — |
| Bergamot | 18.5 | 8.78–11.43 | 81.11–85.55 | 0 |
| Cajeput | — | 0.35–0.41 | 22.78–32.78 | — |
| Caraway | 2–3 | 0.35–0.53 | 48.89–57.78 | 6.6 (extra strong) |
| Cassia | 2 | 9.62–10.17 | 38.89 | 6.7 (flowers) |
| Cedar wood | — | 0.92 | 7.78–12.78 | — |
| Cinnamon | — | 4.91–5.13 | 57.78–65.55 | 25.7 |
| Citronella | — | 3.35–3.43 | 66.67 | — |
| Clove | 12 | 2.35–2.48 | 50–54.44 | 80.5 |
| Copaiva | 80 | — | — | 0 |
| Cubebs | — | — | — | 0 |
| Eucalyptus | — | 0.35–0.52 | 27.78–36.37 | 0 |
| Fennel | 4.4 | — | — | — |
| Geranium, Spanish | — | — | 35.55 | 0 |
| Juniper 1. | — | 1.12 | foreign: 87.78 | 0 |
| Juniper 2. | — | 0.7–0.84 | English: 62.22 | — |
| Lavender, Mitcham | — | 3.14–3.70 | 45–48.89 | 0 |
| " Hitchin | — | 0.52 | 61.11 | |
| " French | — | 4.9–9.2 | 42.22–44.44 | |
| Lemon | 48 | 0.67–1.90 | 74.44 | 23.6 |
| Lemon-grass | — | 2.01–2.25 | 40–45.55 | — |
| Lime | — | 1.41–1.49 | 66.67–72.78 | — |
| Mace | — | 0.67–0.85 | 46.67–50 | — |
| Neroli | 9–11.5 | — | — | 0 |
| Nutmeg | — | 0.19–0.35 | 47.78–50.55 | — |
| Orange, bitter | 50 | 0.38–0.39 | 62.22–66.67 | 6.9 |
| " sweet | 30 | | | |
| Pennyroyal | — | 1.46–4.62 | — | 0 |
| Peppermint, English | 4.5 | 1.83–1.97 | 19.44–21.11 | 0 |
| " American | | 2.71–4.37 | 22.22–34.44 | |
| " Japanese | | 2.22–2.29 | 20–25.55 | |
| Rosemary | — | 0.78–0.88 | 42.78–56.67 | 0 |
| Rue | — | 0.29–0.56 | 66.11–83.33 | — |
| Sage | — | 1.58–7.09 | 69.44 | 0 |
| Sassafras | 20 | 0.28–0.33 | 11.11–31.67 | — |
| Spike lavender | — | — | 38.89–71.22 | — |
| Star anise | 19 | — | — | 79.8 |
| Thyme | — | 1.09–1.91 | 42.22 55.55 | — |
| Turpentine, French | 500 | — | 57.78–81.11 | 0 |
| " rectified | 60 | — | | |
| Verbena | — | 1.20–1.37 | 40–48.89 | — |
| Wintergreen | 9–11 | — | — | 89.4 (artificial) |

*Langbeck's method for the detection of adulterated volatile oils.**—H. W. Langbeck observed that salicylic acid is soluble in volatile oils, but more freely in oils containing oxygen than in those that do not. Thus, for instance, the oils from plants of the labiate family dissolve large quantities; those from umbelliferous plants, with few exceptions, dissolve smaller quantities; and those from plants of the conifer, dipterocarpus, and cassia families very small quantities. In the compilation of the appended table the method of procedure was as follows: Into each of a number of dry flat-bottomed test-glasses, about two inches long and five-sixteenths of an inch in diameter, was placed 0.05 gramme of salicylic acid. Each glass thus prepared was stuck in a flat cork to avoid upsetting and exactly weighed. An oil known to be pure was then added, drop by drop, and shaken until a clear solution resulted, when the increase in weight found gave the relative solubility. The same oil was then mixed with five per cent. and ten per cent. of turpentine oil and each mixture tested.

The essential oils as they come into commerce contain almost always small quantities of water derived from the distillation with steam, and to this moisture is due the formation of more or less hydrogen peroxide, according to the length of time an oil is kept. In order proximately to determine this quantity, and thereby to be able indirectly to judge as to the age of the oil, potassium iodide starch paper is used, prepared from 1 part of starch, 2 of potassium iodide, and 100 of water. The oil to be examined is shaken with an equal quantity of distilled water, and after being allowed to settle completely a few drops of the water are allowed to trickle upon the paper. If the oil is fresh or recently rectified, the paper remains white. An older oil produces more or less intense color, and allows by comparison with oils of which the age is known, after some practice, tolerably certain conclusions to be drawn. Various volatile oils, such as oil of pennyroyal, peppermint (Hotchkiss), bergamot, and turpentine rectified by H. W. Langbeck in June, 1882, and preserved with pieces of fused calcium chloride in glass-stoppered flasks, part in direct sunlight and part in a half-darkened cupboard, did not color the iodized paper; they had preserved their characteristic odors, and the peppermint oil had even acquired a much finer smell.

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* Pharm. Jour. and Trans., 1885.

The oils thus preserved remained during the first twelve months remarkably clear and white; a yellowish turbidity then appeared above the pieces of calcium chloride, which in two years had become communicated to the whole of the oil. It should be mentioned that from time to time these oils were brought into use, so that the flasks were only partially filled.

| Name of oil. | Approximate age. | Turpentine oil added. | Relative solubility. |
|---|---|---|---|
| Angelica | 6 months | — | 1: 48.85 |
| “ | “ | 5 per cent. | 1: 66.75 |
| “ | “ | 10 “ | 1: 101.35 |
| Anise, star | “ | — | 1: 53 |
| “ common | freshly rectified | — | 1: 74.7 |
| “ “ | “ “ | 5 per cent. | 1: 94 |
| “ “ | “ “ | 10 “ | 1: 116 |
| “ “ | 6 months | — | 1: 70 |
| “ “ | “ | 5 per cent. | 1: 81 |
| “ “ | “ | 10 “ | 1: 100.6 |
| Bergamot | freshly rectified | — | 1: 30 |
| “ | “ “ | 5 per cent. | 1: 36 |
| “ | “ “ | 10 “ | 1: 42 |
| “ | one year | — | 1: 17 |
| “ | “ “ | 5 per cent. | 1: 22.5 |
| “ | “ “ | 10 “ | 1: 36 |
| Cajeput | “ “ | — | 1: 10 |
| Calamus | “ “ | — | 1. 15 |
| Caraway | “ “ | — | 1: 85 |
| Cassia | “ “ | — | 1: 7.7 |
| Citronella | “ “ | — | 1: 10 |
| “ | “ “ | 5 per cent. | 1: 19 |
| Cloves | freshly rectified | — | 1: 56 |
| “ | “ “ | 5 per cent. | 1: 68 |
| “ | two years | — | 1: 25.5 |
| Copaiba | freshly rectified | — | 1: 95 |
| Coriander | two years | — | 1: 84 |
| Dill (German) | fresh | — | 1: 15 75 |
| “ “ | “ | 5 per cent. | 1: 20 |
| “ “ | “ | 10 “ | 1: 26.8 |
| “ (English) | one year | — | 1: 12.5 |
| “ “ | “ “ | 5 per cent. | 1: 15 |
| “ “ | “ “ | 10 “ | 1: 20 |
| Eucalyptus | “ “ | — | 1: 13 |

| Name of oil. | Approximate age. | Turpentine oil added. | Relative solubility. |
|---|---|---|---|
| Fennel | one year | — | 1: 31 |
| Ginger grass | " " | — | 1: 14 |
| Juniper wood | " " | — | 1: 130 |
| Lavender (Mont Blanc) | fresh | — | 1: 12 |
| Lemon | 6 months | — | 1: 80.5 |
| " | " | 5 per cent. | 1: 104 |
| " | " | 10 " | 1: 125 |
| Marjoram | two years | — | 1: 17.6 |
| Mustard | fresh | — | 1: 58 |
| Neroli | one year | — | 1: 13.2 |
| Nutmeg | fresh | — | 1: 31 |
| Pennyroyal | " " | — | 1: 25 |
| Peppermint (Hotchkiss) | one year | — | 1: 7 |
| " " | " " | 5 per cent. | 1: 14.6 |
| " " | " " | 10 " | 1: 26 |
| Rosemary | " " | — | 1: 10 |
| " | freshly rectified | — | 1: 12.5 |
| " | " " | 5 per cent. | 1: 17.65 |
| " | " " | 10 " | 1: 24 |
| Thyme | fresh | — | 1: 55* |
| Turpentine | freshly rectified | — | 1: 625 |
| " | three months | — | 1: 540 |
| " | for two years | — | 1: 159 |
| " | exposed to direct sunlight | — | |
| " | partially resinified | — | 1: 94 |
| Valerian | three years | — | 1: 15 |
| Wintergreen | fresh | — | 1: 37.5 |
| Wormwood | one year | — | 1: 17.35 |
| " | " " | 5 per cent. | 1: 40 |
| " | " " | 10 " | 1: 51.85 |

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[*] The oil contained nearly no thymol.

# APPENDIX.

## MANUFACTURE OF LUBRICANTS.

THE object of lubrication is, in brief, the reduction of friction and the preservation of machinery.* However, the limits of this work do not permit us to enter upon this subject, and we, therefore, restrict ourselves to giving approved formulæ for the manufacture of such lubricants of which animal and vegetable fats and oils form the chief constituents.

### *Tallow Lubricants.*

Tallow lubricants yield always good results, only they are somewhat more expensive than others. The consistency of tallow varies so much according to the temperature, as at one time to induce the management of the Austrian State Railroad, where this lubricant is used for locomotives and cars, to use every month one of different composition. But experience proved this of no practical value, so that at present but three varieties are used, one for winter, one for summer, and one for spring and fall. Their composition is as follows:

| Lubricant for— | Tallow. Parts. | Olive oil. Parts. | Old lubricant, or lard, or horse fat. Parts. |
|---|---|---|---|
| Winter | 100 | 20 | 13 |
| Spring and Fall | 100 | 10 | 10 |
| Summer | 100 | 1 | 10 |

(Old lubricant is the residue which remains in the grease-boxes from one lubrication to the other, and is generally of the consistency of lard.)

The lubricant is prepared by melting the fats together, and after heating, with constant stirring, to about 302° F., allowing the mass to cool.

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* The various apparatuses for, and the methods of, testing the viscosity, frictional resistance, etc., of lubricants, have been fully discussed in "Petroleum: Its History, Origin, Production, Physical and Chemical Constitution, Technology, Examination and Uses." By William T. Brannt. Philadelphia, Henry Carey Baird & Co., 1895.

For working on a small scale an ordinary pot is used, and on a large scale a boiler placed in a water bath and provided with a stirring apparatus. The liquid and easily melted fats are first brought into the boiler, and the tallow is finally added.

*Tallow and train-oil lubricant.* Purified tallow 2 parts, train oil 1. It is prepared by melting the tallow in a kettle at as low a temperature as possible, then adding the train oil, and stirring until a homogeneous mass is obtained.

To give this lubricant a sufficient degree of fluidity for use in cold climates, the quantity of train oil has to be increased. This holds good for every other lubricant, the receipts here given being calculated for a moderate climate.

*Sulphur axle grease.* Purified tallow, 2 parts; train oil, 2; sulphur (flowers of sulphur), 1.

Melt the tallow, and after heating to the boiling point of water, add the train oil, mix by stirring, and finally the sulphur. Then keep the whole at the boiling point of water for about ten minutes, withdraw the fire, and stir until the mixture forms a homogeneous, butyraceous mass.

*Booth's lubricant. No. 1:* Purified tallow, 6 parts; palm oil, 12; water, 8; soda, 1.

*No. 2:* Purified tallow, 8 parts; palm oil, 20; water, 10; soda, 1.5.

For both receipts first melt the tallow, and after heating to about 302° F., stir in the palm oil. Dissolve the soda in water in a special vessel, either at an ordinary temperature or with the assistance of heat, and pour the solution in a *thin* stream into the mixture of tallow and palm oil, stirring constantly. When all the soda solution has been added, withdraw the fire, and stir until the mass opposes considerable resistance to the stirrer.

Lubricants prepared according to these receipts are of excellent quality, and are largely used by English railroads. Their consistency may be changed at will by varying the proportions of tallow to palm oil; the the greater the proportion of tallow, the more solid the grease.

*Tallow and neat's foot oil lubricant.* Tallow and neat's foot oil, 100 parts each.

This lubricant was for a number of years used by the Wurtemberg railroads. It is very thickly fluid, and therefore especially suitable for summer use. It is, however, expensive.

*Tallow, colza oil, and soda lubricants. No. 1, for winter:* Tallow, 180 parts; refined colza oil, 120; soda, 20; water, 300.

*No. 2, for spring and fall:* Tallow, 230 parts; refined colza oil, 85; soda, 20; water, 350.

*No. 3, for summer:* Tallow, 260 parts; refined colza oil, 55; soda, 20; water, 340.

*French lubricant of tallow and train oil.* Tallow, 260 parts; train oil, 230; soda, 23; water, 500.

*Tallow and castor-oil lubricant.* Castor oil, 140 parts; lard, 14; tallow, 7.

This lubricant is very useful for fine machines, but too expensive for ordinary purposes.

### *Palm-oil Lubricants.*

Solid lubricants prepared with palm oil are extensively used for greasing wagons, railroad cars, axles of fly-wheels, and other quick-running parts of machines.

Lubricants prepared with palm oil have generally a fine yellow color, varying from deep yellow to orange, which they derive from the unbleached palm oil used.

There are lubricants which contain no other fat but palm oil, but experience has proved that, though cheap, they do not possess the properties demanded from a good article. Their melting-point, as a rule, is too low, and they become too thinly fluid in consequence of the heat developed by the motion of the parts of the machine, which occasions great losses. They are, therefore, not well adapted for greasing axles of wagons and railroad cars.

To make palm-oil grease more thickly fluid, some tallow is generally added, or, where greater fusibility is of importance, a fluid fat, such as train oil or colza oil. By the addition of these the cost of production is considerably increased, but this would seem to be of little importance in view of the improvement in quality, and for rolling stock, where it is almost impossible to save the grease dropping from the axles, such lubricant is actually cheaper, as it lasts much longer.

Palm oil lubricants contain varying quantities of fat, whether palm oil alone is used or in combination with other fats; as a rule, the quantity of fat is between 35 and 50 per cent. of the total weight of the lubricant.

*Palm oil and soda lubricants.* A good serviceable palm-oil lubricant should contain a small quantity of soda (crystallized carbonate of soda). This addition of soda is of great importance, the good quality of the lubricant actually depending on it.

Soda and the alkaline carbonates in general possess the property of forming emulsions with fats, *i. e.*, they convert them into milky fluids. By shaking soda solution with a liquid fat the mixture gradually assumes a white milky appearance. Viewed under the microscope such milky fluid appears colorless, but shows innumerable small globules of fat distributed in the same manner as butter in milk.

The formation of the emulsion has a twofold object: first, to introduce into the composition of the lubricant a large quantity of water (the water used for dissolving the soda), by which means the bulk of the lubricant is considerably augmented without increasing the cost of production. The second object attained by the addition of soda would seem as follows: Palm oil consists largely of palmitin, but beside contains considerable quantities of free fatty acids, this being the case especially with old oil. These fatty acids possess the property of strongly attacking metals, a lubricant of palm oil alone exerting for this reason a very destructive effect. But if the lubricant contains a sufficient quantity of soda, the fat, on the one hand, is converted into an emulsion, or, so to say, enveloped by the soda solution, which reduces the injurious effect upon metals, and, on the other hand, certain free fatty acids seem to possess the property of expelling the carbonic acid from the carbonate of soda, and forming combinations—called soap—with the soda.

The so-called caustic alkalies possess in a high degree the property of saponifying free fatty acids. They withdraw them from the fats and form soaps with them even at an ordinary temperature. Now, by substituting caustic soda for the carbonate, the entire quantity of free fatty acids present is immediately saponified, so that there need be no fear of the metal being attacked in any manner whatever.

Carbonate of soda can be converted into caustic soda by the following simple process. Dissolve the soda to be used in ten times its quantity of water, and boil the solution with as much slaked lime as soda for a few minutes in an iron boiler. Then cover the boiler, and, after the lime has settled on the bottom, draw off the clear fluid.

Solid caustic soda is now found in commerce. Where it can be obtained cheap enough, it can be immediately used for the preparation of the lubricant, one-quarter less being taken than the quantity of soda mentioned.

The method of fabrication is the same as that with soda solution, and requires less time; the solution of caustic soda forming a quicker and more intimate mixture with the fats than soda solution.

*Preparation of palm oil and soda lubricants.* For preparing small quantities (up to 200 pounds) an ordinary boiler bricked in and heated by an open fire suffices. For larger quantities it is advisable to effect the melting process by steam, and the stirring by a suitable mechanical contrivance.

Experience has shown that better palm-oil lubricants are obtained by preparing large quantities at one time. The reason for this is that a more intimate mixture of the ingredients is possible on account of a large quantity remaining fluid for a longer time.

The operation commences by bringing the fat most difficult to melt, generally tallow, first into the boiler, and, after melting, adding the palm oil. The two fats are then thoroughly mixed by stirring, and heated to the boiling point of water.

The soda, or caustic soda, is dissolved in the prescribed quantity of water, and the solution, after heating to the boiling point, added to the melted fat with constant stirring. Should no vessel of sufficient capacity to hold the entire quantity of water required for the solution of the soda be available, the soda is dissolved in a little water, the solution added to the fats, and then gradually the remaining quantity of hot water, or, what is still more suitable, the soda is dissolved in portions, and the solutions added to the fats.

The stirring of the fats mixed with soda solution must be so carried on as to effect as uniform a distribution of the soda as possible. It is best to bring the fat into violent motion, and then add the hot soda solution in a thin stream. Stirring is continued until the mass commences to become viscous, and a sample solidifies completely in a short time.

Lubricants of palm oil and soda are generally not brought directly upon the parts of the machine to be lubricated, but are mostly put in special grease boxes placed immediately over the axle to be greased, and provided below with an aperture through which the lubricant reaches the spot to be greased. In consequence of the heat developed by friction, the lubricant in the grease box partly melts after a few revolutions of the axle, or is at least converted into a sufficiently soft mass to penetrate through the above-mentioned aperture. Lubricants assuming the consistency of soft cream are generally the most suitable.

The composition of the lubricant should vary according to the season of the year in which it is to be used, that for summer requiring more tallow than that for winter. For the latter but little tallow is taken, and a fluid fat, for instance train oil or colza oil, is sometimes substituted for a portion of it. Many manufacturers make, besides, a special lubricant for spring and fall, which can be recommended for countries with very hot summers and very cold winters.

Below receipts for the preparation of palm oil and soda lubricants are given. It may be again repeated that the melting point of the lubricant can readily be altered by changing the proportions of the fat.

*Yellow lubricant of palm oil and soda. No. 1, for winter:* Tallow, 750 parts; palm oil, 500; sperm oil or colza oil, 70; soda, 228; water, 2600.

*No. 2, for spring and fall:* Tallow, 800 parts; palm oil, 500; sperm oil or colza oil, 55: soda, 220; water, 2500.

*No. 3, for summer:* Tallow, 900 parts; palm oil, 500; sperm oil or colza oil, 44; soda, 216; water 2450.

The lower the temperature, the less fat and the more water may be used, the proportions being the reverse in summer.

Train oil may be advantageously used as a substitute for the more expensive sperm oil or colza oil, but it has to be previously subjected to a special treatment.

Train oil exposed to a moderately low temperature—about 23° to 20° F.—separates a considerable quantity of solid fat; the portion which does not congeal being much more thinly fluid than the original train oil, and is especially suitable for an addition to the above lubricants. The congealed portion can be used either in the manufacture of soap or of special lubricants.

*Yellow lubricant of palm oil, soap, and soda. No. 1, for winter:* Palm oil, 160 parts; soap, 50; soda, 16; water, 540.

*No. 2, for summer:* Palm oil, 160 parts; soap, 50; soda, 20; water, 360.

Melt the palm oil first, and then add the soap. When the latter is melted add the soda, previously dissolved in 40 parts of hot water for No. 1, and in 60 parts for No. 2. Then mix the rest of the water with the mass constantly kept hot. After all the water has been added, the contents of the kettle are poured into a vat and stirred until the commencement of congelation.

These lubricants can also be prepared by melting the fat and soap together in a kettle and mixing them by stirring. The soda is dissolved in the required quantity of water in a second boiler, the solution heated to the boiling point and intimately mixed by constant stirring with the melted fat in a third vessel. Stirring should be continued as long as the consistency of the fluid permits.

*American palm oil lubricant.* *A.* Tallow, 150 parts; palm oil, 100; soda, 25; water, 160.

*B.* Tallow, 100 parts; palm oil, 160; soda, 35; water, 300.

*French palm-oil lubricant.* *A.* Tallow, 380 parts; palm oil, 125; colza oil, 65; soda, 25; water, 420.

*B.* Tallow, 280 parts; palm oil, 100; colza oil, 70; soda, 20; water, 500.

*Belgian palm-oil lubricant.* *A.* Palm oil, 210 parts; soap, 85; soda, 15; water, 700.

*B.* Tallow, 750 parts; palm oil, 380; colza oil, 200; soda, 50; water, 1300.

These lubricants, besides being cheap and rendering excellent service, have the advantage of a combination of three fats, viz., tallow, palm oil, and colza oil.

*Axle-grease for very heavy wagons.* *No. 1, for winter:* Tallow, 420 parts; palm oil, 840; soda, 140; water, 4200.

*No. 2, for summer:* Tallow, 420 parts; palm oil, 490; soda, 35; water, 2300.

The above lubricants are calculated for a low temperature in winter and a hot one in summer. For a moderate winter climate, the quantity of soda may be somewhat reduced, and that of palm oil increased.

*Axle grease for heavy wagons.* Palm oil, 210 parts; tallow, 85; soda lye, 65; water, 920.

Melt the palm oil and tallow together, and after making the mixture homogeneous by stirring, add the soda lye. The latter is prepared by dissolving soda in sufficient hot water for the cooled fluid to show 20° or 21° Bé. After adding and stirring in the soda lye, the water is added, and the whole stirred to a homogeneous mass, which is ladled into vessels and allowed to congeal.

*Grease for wooden machinery.* Tallow, 30 parts; palm oil, 20; train oil, 10; graphite, 20.

After melting the fats at a moderate heat, intimately mix the graphite, previously pulverized and elutriated, with the mass by stirring.

### *Lead-soap Lubricants.*

Lead salts have the property of yielding on saponification with fats or fixed oils quite solid combinations called lead soaps. They are hard in the cold and viscous at an ordinary temperature, but on heating by friction acquire the requisite degree of fluidity.

To prepare these lubricants a solution of basic acetate of lead or sugar of lead is combined with the respective quantity of fat. The lead solution is prepared as follows: Sugar of lead, 10 parts; litharge, 10, and soft water 110, are boiled for one and a half to two hours, stirring frequently. The mass is then allowed to rest, and the clear fluid drawn off and restored to 100 parts by the addition of water. It is then heated to from 145° to 165° F., and immediately mixed with ordinary fat—colza oil, lard, and sometimes neat's foot oil—as, for instance : Sugar of lead solution, 100 parts; colza oil, 80; lard, 80.

The preparation thus obtained should show a uniform gray color, and after melting solidify at from 100° to 122° F.

The principal constituent of so-called anti-friction lubricants, the preparation of which is kept a secret, is nothing but lead soap prepared according to the above formula or one varying but little from it. On account of the high melting-point of this lubricant it is better adapted for quickly-running axles (railroad wheels, fly-wheels, etc.) than for ordinary wagons.

The preparation of lead-soap lubricant can be considerably modified by the following process: Sugar of lead, 20 parts; litharge, 10; water, 225; vinegar, 1.

The water and vinegar are brought into a vat provided with a well-fitting lid. Two linen bags, one containing the sugar of lead and the other the litharge, are suspended in the fluid, and, after putting on the lid, the whole is allowed to stand one week. The clear solution of acetate of lead is then drawn off into a vat and mixed with 500 parts of colza oil and 750 parts of lard, previously melted together, and heated to 370° F. The whole is then thoroughly stirred and allowed to stand several days. In four, and sometimes even in two days, the mass is congealed and can be at once used.

*Axle grease from lead oleate. Palmer's patent wagon grease* consists principally of lead oleate, and is therefore a lead soap resembling in consistency the sticking-plaster of the drug stores.

In the manufacture of stearin candles the crude fatty acids are subjected to a strong pressure, whereby oleic acid, containing some stearic and palmitic acids in solution, runs off. The crude oleic acid deposits at a low temperature a large portion of the dissolved solid fatty acids in the form of crystals, and is brought into commerce as a yellow to brown fluid, generally of a disagreeable odor.

To prepare lead soap with oleic acid, the latter is brought into a kettle bricked in so that its contents cannot come in contact with the flame, and provided with a well-fitting lid to extinguish the flame in case the oleic acid should ignite.

Heat the oleic acid to the boilng point and then add, with constant stirring, *very finely ground* litharge to the boiling fluid, one-fifth to one-fourth by weight of the oleic acid of litharge being generally used.

After adding the litharge, which is best allowed to fall into the oleic acid through a narrow tube, stirring is continued for 20 to 40 minutes, and then the temperature gradually moderated. The portion of litharge not dissolved by the oleic acid deposits on the bottom of the boiler, where it remains for the next operation. The clear fluid is drawn off into sheet-iron vessels and allowed to congeal.

For quick-running axles, which develop considerable heat, this lubricant soon acquires the requisite degree of fluidity; for slow-running axles it is too viscous.

The melting point of this very useful lubricant is best lowered during its preparation by the addition of cheap fats, such as horse fat or train oil. This is effected by bringing the hot lubricant, prepared as above, into a vat containing the fat or train oil, and combining them by stirring. The lower the melting-point of the lubricant is to be, the more fat has to be added, though, generally speaking, 20 to 40 parts of fat suffice for 100 parts of lead oleate.

### *Actual Soap Lubricants.*

Soap lubricants are prepared with ordinary soft soap (a combination of fatty acid with potash), or by means of fats and potash, which combine to the already mentioned emulsions. These lubricants, though very serviceable for some purposes, are but little used, since it may readily happen that, when carelessly prepared, a portion of the potash lye is not fixed, and by attacking the metals, causes great damage.

*Chardon's soap lubricant.* Potash soap, 10 to 50 parts; lye or water, 90 to 50.

This peculiar lubricant is claimed by the inventor to be much cheaper than oil, and to counteract the wearing of the journal, which if verified would of course be valuable qualities. The varying quantities of soap and water refer to the different seasons of the year. But the use of lye for dissolving the soap seems to us hazardous, since caustic alkalies strongly attack the metals and mixtures of metals of which the parts of a machine are generally constructed. Properly prepared soap contains no free fatty acid, the caustic potash or caustic soda added in the form of lye acting only upon the metals, so that in a short time they appear as coated with rust. That Chardon's lubricant is of little value is shown by the fact that, according to the receipt, it is entirely indifferent whether lye or water is used. Every one who possesses but slight chemical knowledge knows that there must be a great difference between a lubricant consisting simply of soap water and one which contains soap and lye.

*Lubricant of tallow, oil, and soap.* Tallow, 420 parts; olive oil, 360; potash, 60; water, 650.

Dissolve the potash in water, heat the solution to the boiling-point, add the entire quantity of fat at one time, and increase the fire so that the whole remains fluid. The boiling is then continued, with constant stirring, until complete saponification is indicated by the mass becoming thickly fluid, and a sample can be drawn into threads on cooling. The product thus obtained is chemically a solution of potash very rich in water mixed with an excess of fat, and may be designated as an emulsive lubricant in the actual sense of the word.

*Grease for pleasure carriages. A.* Tallow, linseed oil, and common resin, each 500 parts; caustic soda lye, 315.

*B.* Tallow, 500 parts; linseed oil, 450; common resin, 500; caustic soda lye, 500.

These lubricants form, after thorough stirring, firm salve-like masses of a yellow color, which are easily and uniformly applied to the axles and do good service. Melt first the resin and then add the tallow and linseed oil. After combining them to a homogeneous mixture, pour in the soda lye in portions. The soda lye should be of moderate strength; the lubricants can be made firmer by increasing the caustic soda contained in a certain quantity of soda lye.

Chemically these lubricants, which are rather expensive, consist of a mixture of ordinary soap, resin-soap, and emulsion of fat.

### *Caoutchouc Lubricants.*

*Doulon's caoutchouc lubricant.* Train oil, 200 parts; caoutchouc, 20. Heat the train oil in a kettle to the commencement of decomposition, which is indicated by an ebullition similar to that of boiling, and the development of a disagreeable odor. Then add gradually the caoutchouc in small pieces, stirring the mass vigorously after each addition.

On acconnt of the high price of caoutchouc, this lubricant is not available for general purposes, and besides, lubricants can be prepared which are much cheaper than Doulon's, and are at least equal to it.

*Caoutchouc lubricants for belts.* Caoutchouc, 20 parts; linseed oil, 1000.

Melt the caoutchouc together with the same quantity of linseed oil, and as soon as the mixture commences to evolve vapors stir in 20 parts of linseed oil. Later on add the rest of the linseed oil in portions of 100 parts each.

*American caoutchouc lubricant.* Caoutchouc, 4 parts; oil of turpentine, 8; colza oil, 144; soda, 16; glue, 4; water, 50.

Dissolve the caoutchouc in the oil of turpentine. Boil the colza oil after adding the soda and water, and compounding it with the glue, and stir the solution of caoutchouc into the homogeneous mass.

The expression, "solution of caoutchouc," is, strictly speaking, wrong, since only a small portion is actually dissolved, the remainder only swelling up. For this reason the mass must be long and continuously stirred in order to obtain it homogeneous.

*Adhesion lubricant from caoutchouc.* Caoutchouc, 36 parts; oil of turpentine, 72; glue, 10; tallow, 80; soda, 72: water, 900.

This lubricant, which has a rather peculiar composition, is prepared as follows: Dissolve the caoutchouc in the oil of turpentine in a special vessel at from 300 to 350° F. Melt the tallow in another vessel, add the pulverized soda, and finally, with constant stirring, the water. When all is formed to a homogeneous mass, stir in the caoutchouc solution, and allow the whole to cool.

*Caoutchouc and gutta-percha lubricant.* Caoutchouc and gutta-purcha, each 50 parts, oil of turpentine, 100; tallow, 1000.

Dissolve the caoutchouc and the gutta-percha in the oil of turpentine, heat quite strongly, and add the tallow in portions. The addition of caoutchouc and gutta-percha makes this lubricant very expensive.

*Caoutchouc wagon grease.* Palm oil, 20 parts; train oil, 100: caoutchouc, 2; litharge, 2; sugar of lead, 2.

Heat the comminuted caoutchouc together with the train oil to about 480° F., add to the filtered mass the litharge and sugar of lead, heat again for one hour at 480° F., and, finally, stir the palm oil into the hot mass.

*Caoutchouc and fat lubricant.* Caoutchouc, 5 parts; palm oil, 100: colza oil, 100; tallow, 50.

Dissolve the caoutchouc in the colza oil by strong heating, filter, and combine the solution with the solid fats.

### *Other Solid Lubricants.*

In the following we give receipts for the preparation of some lubricants which do not belong to any one of the preceding groups. Such receipts as yielded unfavorable results, or the composition of which indicated that they were recommended by persons unacquainted with the subject, have been omitted.

*Asphaltum axle grease.* Asphaltum, 32 parts; black pitch, 8; petroleum, 8; litharge, 8; water, 80.

Melt the asphaltum and pitch together in a kettle, add the petroleum, and, after the mass has acquired a uniform fluidity, stir in the litharge. Finally, add the water in small portions, and stir until the mass is homogeneous. The asphalt and pitch give to this lubricant a lustrous black color and a peculiar bituminous odor. The consistency of the lubricant can be decreased or increased by the quantity of petroleum added.

*Naphthaline grease.* Naphthaline, 100 parts; colza oil, 50 to 100.

The naphthaline, which is a crystalline hydrocarbon obtained from coal tar, is melted and more or less colza oil stirred in, according to the consistency the lubricant is to show. Under naphthaline (and the same holds good as regards paraffine) must not be understood the pure article, which is rather expensive. Absolute purity is not required for our purposes, the crude product answering all demands.

*Rosin lubricant.* Common rosin, 100 parts; rosin oil, 50; lard, 300.

Melt the rosin and rosin oil together, and stir the lard into the fluid mass.

*Graphite axle grease.* Tallow, 36 parts; lard, 9; palm oil, 9; graphite, 2.

Graphite, which is of a steel-gray color, gives to this lubricant a peculiar black-gray color.

Graphite axle grease is a very usedful composition, and is much employed for greasing axles of wagons in England and Belgium.

*Graphite lubricant for quick-moving axles.* Tallow, 100 parts; graphite, 100.

This lubricant is especially adapted for circular saws, ventilators, etc., and generally for shafts revolving with great velocity without great load.

## *Patent Axle Greases.*

In the preparation of the so-called patent axle greases, residues of rosin oil, paraffin oil, of petroleum and mineral oils in general are used. Lime in the form of calcium hydrate is employed as a cementing (saponifying) agent, while heavy spar (barytes) as well as elutriated kaolin serve as filling agents.

Calcium hydrate is prepared as follows: Fresh burnt lime of good quality and free from sand is spread out and gradually moistened with water (50 quarts to 220 lbs. lime) by means of a watering pot provided with a rose. By this treatment the lime crumbles to a fine white powder, which is passed through a fine sieve and kept for use in a well closed barrel or box.

*a. Patent axle grease in the cold way.* It is advisable to prepare never more than at the utmost two hundred weight of axle grease at one time, since the smaller the bulk of the mass the better it can be worked.

The vats required for the preparation of the respective axle greases are obtained by simply sawing a suitable large oil barrel in half.

1. *Black patent axle grease.* Petroleum residue, 25 lbs.; dark paraffin oil, 15 lbs.; heavy spar, 20 lbs.; calcium hydrate, 10 lbs.; crude rosin oil, 15 lbs.

Bring the petroleum residue into a vat and mix the paraffin oil with it; finally stir in the heavy spar and the calcium hydrate in small portions.

When the whole is thoroughly mixed, pass it with constant stirring through a sieve into another vat, and stir again thoroughly. Now add with constant stirring the crude rosin oil in a thick jet—one workman stirring, while another pours in the rosin oil—and continue stirring until the mass thickens, which, as a rule, takes place in a short time. Stirring is then interrupted and the grease is ready for use.

It may be remarked that the quantity of crude rosin oil required cannot be accurately given, since the saponifying power of the oil varies. It is therefore advisable to determine the quantity of crude rosin oil required by an experiment on a small scale.

Patent axle grease of good quality should be of the consistency of butter, and must not be sticky.

2. *Green patent axle grease.* Green paraffiin grease,* 40 lbs.; heavy

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* Residue obtained in the manufacture of paraffin.

spar, 20 lbs; calcium hydrate, 10 lbs.; crude rosin oil, 14 lbs. Prepared in the same manner as No. 1.

3. *Yellow patent axle grease.* Reddish yellow paraffin oil, 40 lbs.; heavy spar, 20 lbs.; calcium hydrate, 10 lbs.; crude rosin oil, 14 lbs. Prepared like No. 1.

4. *Blue patent axle grease.* Dark paraffin oil with a strong blue fluorescence, 40 lbs.; heavy spar, 20 lbs.; calcium hydrate, 10 lbs.; crude rosin oil, 14 lbs. Prepared like No, 1.

*b. Patent axle grease in the warm way.* In preparing patent axle greases in the warm way a basis-mass is required, which is prepared as follows: Bring into an iron kettle 200 lbs. of crude rosin oil, and with constant stirring and heating, 160 lbs. of calcium hydrate. Then boil the mass with contant stirring until it is thoroughly homogenous and runs from the stirrer syrup-like in long, viscous threads. Heating is then interrupted and the mass stirred until almost cold.

1. *Black patent axle grease.* Basis-mass, 50 lbs.; petroleum residue, 70 lbs.; crude rosin oil, 10 lbs.

The basis-mass and the petroleum residue are brought into an iron kettle and moderately heated and intimately mixed by vigorous stirring. The mixture is then poured through a sieve into a vat, and, when cold, stirred for a few minutes. The crude rosin oil is then poured in with constant stirring, and stirring continued until the mass thickens.

2. *Deep dark-green patent axle grease.* Blue or green mineral oil, 30 lbs.; finely sifted calcium hydrate, 32 lbs.; residuum continuing asphalt, 28 lbs.; cold, heavy, blue rosin oil, 30 lbs.; crude wood tar oil, ½ lb.

Bring the blue or green mineral oil and the calcium hydrate into an iron kettle, and heat with constant stirring to boiling. Then add the residuum containing asphalt, stir vigorously until all is dissolved and a homogeneous mass has been formed. Bring the warm mass into a vat, and stir until it is lukewarm. Then add the rosin oil and wood tar oil, and stir until the mass commences to solidify. For residuum that obtained in the distillation of petroleum may be used.

3. *Green patent axle grease.* Basis-mass, 50 lbs.; green paraffin grease, 70 lbs.; crude rosin oil, 20 lbs. Prepared like No. 1.

4. *Yellow patent axle grease.* Basis-mass, 50 lbs.; yellow paraffin oil, 70 lbs.; crude rosin oil, 20 lbs. Prepared like No. 1.

5. *Blue patent axle grease.* Basis-mass, 50 lbs.; blue paraffin oil, 70 lbs.; crude rosin oil, 20 lbs. Prepared like No. 1.

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6. *Bluish patent axle grease.* Thin rosin oil, 40 lbs.; paraffin oil, 80 lbs: rectified blue rosin oil, 160 lbs.; basis-mass, 180; heavy spar, 60 lbs.

The "basis-mass" is prepared as follows: Bring into an iron kettle 200 lbs. of thin rosin oil, and with constant heating and vigorous stirring add 160 lbs. of calcium hydrate. When saponification is complete, and no more lumps are observed in the mass, and the latter has acquired the consistency of syrup, heating is interrupted, and stirring continued until the mass is cold. This basis-mass is kept for preparing bluish axle grease. For use it is heated and vigorously stirred.

*According to another method*, bluish axle grease is prepared as follows: Heat in an iron kettle calcium hydrate, 150 lbs; blue mineral oil, 50 lbs.; tar oil, 50 lbs., until the mass is perfectly homogeneous.

*Patent axle grease from rosin and paraffin oil without saponification by lime.* Dark paraffin oil, 70 lbs.; crude rosin oil, 25 lbs.; soap powder, 5 lbs.; brown American rosin, 10 lbs.

Convert rosin soap of good quality into fine powder by planing it to shavings, drying them, and finally pounding in a mortar.

Bring into a capacious kettle, which must not be filled more than half, the dark paraffin oil,* the crude rosin oil and the soap powder and heat moderately. The mass commences to foam and rise in the kettle, which must be prevented as much as possible by vigorous stirring. The formation of foam lasts until all the soap powder is dissolved. When this is the case the mass subsides, and shows a smooth surface. At this stage add with constant stirring the rosin, and continue stirring until it is dissolved. Now bring the grease into a vat provided with a stirring apparatus, and continue stirring until it is nearly solidified.

This patent axle grease may be filled by mixing it with 10 to 20 per cent. of elutriated alumina, heavy spar or talc. The mixture is effected by gradually adding one of the above mentioned earthy substances to the grease while in the mixing vat, or still better by allowing the respective filling material to fall through a sieve upon the surface of the grease while the latter is being stirred in the mixing vat.

*Patent axle grease in the cold way according to Thein.* Fresh fat lime, 60 lbs.; rectified heavy tar oil, 100 lbs.; rosin oil, 100 lbs.

Slake the lime gradually to a thin fat paste and, while boiling hot, add the tar oil and mix by vigorous stirring. When the mass, by constant stirring, is cooled to about 116° F. an intimate mixture of the lime with the oil takes place, in consequence of which every particle of water is separated. When this is the case, and the mass is cooled to 77° F., the rosin oil is stirred in. The resulting product, when cold, is of the consistency of butter, has a greenish-blue color, does not run off from the axle, is very durable, and possesses all the properties of an excellent axle grease.

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* Petroleum residue may be used instead of paraffin oil.

*Patent axle grease in the warm way, according to Cruse.* Wool fat, 150 lbs.; lime meal, 36 lbs.; dark paraffin oil, 300 lbs.; caustic lye of 42° Bé, 17½ lbs. Heat the wool fat in an iron kettle until it is entirely melted, and then introduce with constant stirring 150 lbs. of dark paraffin oil. When the mass commences to boil bring, with constant stirring, one half of the quantity of lime meal, shovelful by shovelful, into the kettle. Then add 150 lbs. more of paraffin oil and gradually the remainder of the lime meal, and finally introduce carefully the caustic lye.

However, before the entire quantity of paraffin oil has been added, saponification must have taken place, which is recognized by the evolution in rapid succession of large bubbles upon the surface of the grease, which just as rapidly burst. When the grease is of proper consistency it is finished. It is allowed to cool off in the kettle, and before becoming entirely cold is brought into barrels.

The caustic lye required for this grease is prepared by dissolving potash, if possible, in an equal volume of warm water and when cold bringing the lye to 42° Bé. by the addition of water.

*Blue, yellow, and black patent axle greases, according to Sonnenschein and Leus.* A basis-mass is prepared as follows: Rosin oil, 100 lbs.; lime slaked to powder, 40 lbs. Bring the rosin oil into an iron kettle, stir in the lime powder, and then heat the mixture, with constant stirring, until it forms a homogeneous paste free from lumps, which runs like syrup from the stirring paddle.

With the assistance of this basis-mass the following patent axle greases are prepared.

1. *Blue patent axle grease.* Crude rosin oil, 500 lbs; lime slaked to powder, 2 lbs.; basis-mass 10 to 12 lbs. Boil the rosin oil with the lime powder in an iron kettle for one hour, allow to cool somewhat, then draw off the oil from the sediment, and while the oil is still warm introduce the basis-mass. Stir until the grease is of the consistency of butter and shows a blue color.

2. *Yellow patent axle grease.* Add to the blue grease described

above 6 per cent. of turmeric solution prepared by boiling 1 part turmeric with 20 parts caustic lye of 25° Bé.

3. *Black patent axle grease.* Add to 100 lbs. blue patent axle grease 2 lbs. lamp-black triturated with rosin oil.

*Dr. Herrburger's axle grease.* As raw materials are employed petroleum residuum or paraffin grease as well as heavy viscous coal tar oil.

*First method.* Colza oil, 20 lbs.; wool fat free from water, 80 lbs.; fresh, thoroughly burnt lime, 10 lbs.; water, 15 to 20 lbs.; petroleum residuum, paraffin grease or heavy, viscous coal tar oil, 1000 to 1200 lbs.

An iron kettle of double the capacity of the quantity of material to be boiled in it has to be used. Introduce the colza oil and 10 lbs. of the wool fat and allow the latter to melt in the oil, keeping up a moderate fire. In the meanwhile bring the lime into a wooden vessel and slake it with the quantity of water given above. When the wool fat is melted bring the slaked lime into the kettle, increase the fire somewhat, and boil with constant stirring until a thick froth is formed and the mass sticks to the bottom of the kettle. To prevent the mass from scorching it must be constanty stirred.

Now add gradually, in portions of about 10 lbs., 70 lbs. of wool fat previously rendered liquid in another kettle, and boil with a moderate fire until the small lumps formed have combined to a perfectly homogeneous mass.

When the mass is cooled off to luke warm, the petroleum residuum, paraffin grease or coal tar oil is added and the whole boiled until perfectly homogeneous.

To the grease prepared in this manner 10 to 20 per cent. elutriated alumina, heavy spar or talc may be added, as described on p. 650.

*Second method.* Petroleum residuum paraffin grease, or heavy viscous coal tar oil, 90 lbs.; wool fat free from water, 10 lbs.; caustic soda lye of 40° Bé., 10 lbs.; brown American rosin, 10 lbs.

Melt together with constant stirring over a moderate fire, the petroleum residuum, paraffin grease or coal tar oil and the wool-fat. Bring the mixture into a woolen vat and, when lukewarm, stir in the caustic soda lye, whereby the mass becomes solid. After reposing for 12 hours the grease is returned to the kettle and the rosin combined with it under constant stirring. The product, when cold, is a beautiful, smooth axle grease of great fatness.

If the grease is to be filled, stir in at the stage when the mixture of oil and wool fat is brought from the kettle into the wooden vat, 10 to 20 per cent. of elutriated alumina, talc or heavy spar, allowing the respective substance to fall through a sieve upon the surface of the grease, the latter being vigorously stirred.

*Filling masses for axle grease.* I. Water, 150 gallons; calcined soda, 40 lbs.; rosin in small pieces, 200 lbs.; wool fat, 40 lbs.; dark paraffin oil, 100 lbs.

Dissolve the soda in the water by boiling, then gradually introduce the rosin and boil, with constant stirring, until the latter is dissolved. Now add the wool fat and, when this is dissolved, the paraffin oil. Continue boiling with constant stirring until a homogeneous mass is formed. The final product is *yellow filling mass.*

*Blue filling mass* is obtained by adding to the yellow product a small quantity of Paris blue.

By substituting green paraffin grease for the paraffin oil, *green filling mass* is obtained, and by mixing some coal tar with the yellow product, *brown filling mass.*

This filling mass is used by dissolving it in the paraffin oils to be used in the preparation of axle grease.

II. Water, 50 gallons, calcined soda, 40 lbs.; slaked lime, 20 lbs.; American rosin, 100 lbs.; cocoanut oil, 2 lbs.; paraffin oil, 40 lbs.

Dissolve the soda in the water by boiling, then add the lime and boil until dissolved. Now introduce the rosin, cocoanut oil, and paraffin oil previously melted together, and boil until thorough combination has been effected. Transfer the mass to a vat and allow it to cool, stirring occasionally. This filling mass is used by mixing it with the oils to be employed in the preparation of the axle grease, and then adding the slaked lime or the " basis-mass."

4. *Preparation of axle grease direct from rosin without rosin oil.* Water, 100 gallons; calcined soda, 40 lbs.; slaked lime, 20 lbs.; American rosin in small pieces, 20 lbs.; wool fat, 100 lbs.; paraffin oil, 200 lbs.

Dissolve the soda in the water by boiling, then add the lime and continue boiling until the latter is dissolved. Now introduce the rosin, wool fat and paraffin oil previously melted together, and boil until thorough combination is effected. Bring the mass into a vat, and let it cool, stirring it occasionally.

Should the axle greasee thus prepared be too thin mix with it 5 to 8 per cent. of heavy spar to the proper consistency.

By adding to the soda solution enough finely powdered turmeric to impart to it a beautiful yellow color *yellow axle grease* is obtained.

*Blue axle grease* is obtained by coloring the finished grease, while in the kettle, blue with Paris blue.

*Green axle grease* is obtained by substituting green paraffin grease for the paraffin oil.

*Brown axle grease* is obtained by mixing the yellow grease with coal tar.

5. *Manufacture of axle grease from pinolin or light rosin oil. a. Yellow axle grease.* Pinolin free from water, 200 lbs.; slaked lime, 150 lbs.; reddish paraffin oil, 300 lbs.; crude heavy rosin oil, 300 lbs.

Heat the pinolin in a kettle over a moderate fire and gradually stir in the lime, whereby the pinolin acquires a pale yellow color. Now remove the fire from under the kettle and introduce the paraffin oil. When this is thoroughly mixed with the pinolin and lime, add the heavy rosin oil and stir until the mass commences to thicken.

*b. Blue axle grease.* Pinolin free from water, 200 lbs.; slaked lime, 150 lbs.; dark paraffin oil, 300 lbs.; crude heavy rosin oil, 300 lbs.

Mix the pinolin and lime in a kettle over a moderate fire and add to the mixture first the paraffin oil, next the rosin oil, and stir until the mass commences to thicken.

6. *Manufacture of axle grease from naphtha residuum.* Rossmässler recommends the following process: Naphtha residuum, 120 to 160 lbs.; tallow, 20 lbs.; rosin, 20 lbs.; soda lye of 40° Be., 30 lbs.

Mix the naphtha residuum, tallow and rosin and melt them together over a moderate fire. When the homogeneous mass has been cooled down to at the utmost 104° F. add, with constant, vigorous stirring, the soda lye in a fine stream. The mass must be constantly stirred until it commences to get viscous and cold. It is then brought into small barrels or boxes in which it solidifies to a homogeneous axle grease of a greenish brown color.

7. *Axle grease with the use of infusorial earth.* Palm oil, 100 lbs.; rosin oil, 40 lbs.; caustic soda lye of 16° Bé., 50 lbs.; paraffin oil, 50 lbs.; finely sifted calcined infusorial earth, 10 lbs.

Melt the palm oil and rosin oil in a kettle, saponify with the lye and add, with vigorous stirring, the paraffin oil. While stirring add the infusorial earth, and continue stirring until the mass is homogeneous and commences to get cold.

For summer use this grease is too soft and hence somewhat more palm oil has to be used.

### *Manufacture of French Patent Axle Greases.*

*Preparation of the basis-mass.* The basis-mass for the blue, yellow and black axle greases prevailing in France is prepared as follows:

Burned lime, 80 lbs.; rosin oil, 100 lbs.

The lime is brought into an open vessel, best a shallow sheet-iron pan, and gradually slaked to a fine powder with one-fourth its quantity of water, the operation being accelerated by frequent stirring with a paddle. When cold the lime powder is passed through a fine flour sieve. Special attention must be paid to the preparation of this lime powder, since the quality of the "basis-mass" depends upon it. It should be fine and floury without being sandy.

Heat in a kettle the above-mentioned quantity of rosin oil until no more vapors are evolved. Then add the slaked lime and stir the mixture until a homogeneous paste free from lumps has been formed which is detatched with difficulty from the stirring paddle.

The fire is now moderated and immediately withdrawn when the particles of water have been removed and intimate saponification has been effected.

With the use of this basis-mass the various kinds of axle grease are prepared.

*Blue axle grease.* Basis-mass, 60 lbs.; rosin oil, 360 lbs. Pour the rosin oil upon the basis mass and vigorously stir until the mass thickens.

For cheaper axle grease stir into the cold grease 20 to 50 per cent. of talc.

*Yellow axle grease.* This is prepared in exactly the same manner as the blue grease, but it is colored yellow with turmeric. The coloring matter is boiled in caustic potash lye of 22° to 24° Bé. (1 lb. of turmeric to 18 lbs. of lye) until dissolved. Six to eight parts of this turmeric solution suffice for 100 parts of grease.

*Black axle grease.* This also is prepared like the blue grease, but is colored black with lamp black triturated with rosin oil. For 100 lbs. of grease about 1½ to 2 lbs. of lamp-black are required.

*Ordinary French axle grease.* I. Red-brown American rosin, 100 lbs.; rendered tallow, 100 lbs.; soda lye of 1.160 specific gravity, 100 lbs.; linseed oil, 100 lbs.

Melt the rosin and the tallow in a kettle over a moderate fire and then add, with vigorous and constant stirring, the soda lye. When the mass ceases to rise, add the linseed oil, boil for fifteen minutes longer and, pour the mass through a cotton filter into a clean vessel in which it is allowed to cool. The product is a beautiful lemon-yellow, granular grease of the consistency of butter and does not gum.

II. Palm oil, 30 lbs.; tallow, 12 lbs.; soda lye of 20° Bé., 9 lbs.: soft water, 130 lbs.

Melt the palm oil and tallow in a kettle, then add, with constant stirring the soda lye and water, and boil the mass for about one hour, stirring constantly. Pour the grease into wooden vessels.

*French axle-grease specialties:* I. Fine lime meal, 50 lbs.; blue rosin oil, 150 lbs.; pale tar oil or pure paraffin oil, 150 lbs.; paraffin oil, 400 lbs.; finely pulverized talc, 400 lbs.

Into a capacious kettle bring first the lime meal, and add gradually the blue rosin oil, stirring until saponification takes place. When the mass no longer boils, in consequence of the slaking of the lime, add with vigorous stirring the tar oil, and, if saponification should be incomplete, add 5 to 6 lbs. of caustic lye of 20° to 25° Bé. Now slightly heat, if necessary, the entire mass, and add gradually the paraffin oil, stirring constantly. If the grease is to be filled stir in 400 lbs. of talc, and color it as desired, using for *black* lamp black triturated with rosin oil, for *blue* ultramarine triturated or boiled with lye of 20° Be'.; for yellow, palm oil or naphthaline triturated with rosin oil.

For summer use the grease is made somewhat thicker than for winter.

In some factories the lime meal used for this grease is advantageously prepared as follows: Fresh burnt lime of a good quality is spread upon the floor and moistened with water by means of a watering pot until it crumbles to powder. The meal is then sifted to free it from sand. The meal thus prepared is brought into the kettle and stirred to a paste with rosin oil. If saponification does not take place in a short time, the process is accelerated by heating the contents of the kettle. In all other respects the process is the same as given above.

II. Fresh fat lime, 300 lbs.; rectified heavy tar oil, 500 lbs., or paraffin oil, 800 lbs.; heavy tar oil, 300 lbs.; rosin oil, 500 lbs.; finely pulverized talc, 25 lbs.

The lime is very gradually slaked to a thick paste, and while the latter is still boiling hot, the rectified heavy tar oil, or the parffin oil, is added, with constant stirring. At 68° to 75° F. an intimate combination of the lime with the oil takes place, in consequence of which the particles of water are separated. The water is carefully removed from the thin paste and the latter allowed to cool to 64° F. The heavy tar oil is then added, and, when the whole is thoroughly mixed, the rosin oil.

The finished grease may be filled, 25 lbs. of finely pulverized talc being used for every 100 lbs. of grease. The talc is best mixed with the last two oils, and the mixture gradually stirred into the mass.

### *Manufacture of Belgian Axle Grease.*

*In the cold way.* I. Lime meal, 60 lbs.; blue rosin oil, 200 lbs.; pale light rosin oil, 200 lbs.; paraffin oil, 500 lbs.; finely-pulverized talc, 500 lbs.

Bring the blue rosin oil together with the lime meal into a kettle, combine them intimately by stirring, and then add, with vigorous stirring, the light rosin oil. When the whole is thoroughly combined add, with vigorous stirring, the paraffin oil, and finally the pulverized talc.

The grease is colored black with lamp black triturated with rosin oil.

For summer use the grease is made some what thicker than for winter.

II. Lime meal, 100 lbs.; paraffin oil, 100 lbs.; thin rosin oil, 30 lbs.; thick rosin oil, 50 lbs.; wool fat, 20 lbs.

The paraffin oil is mixed with the burnt lime slaked to a powder, the thin rosin oil is then added with constant stirring and finally the thick rosin oil, stirring being continued until intimate combination has been effected.

The qualitv of this grease is improved by adding to the paraffin oil the above-mentioned quantity cf wool fat and taking 20 lbs. less of each of the two rosin oils.

III. Lime meal, 10 lbs.; blue rosin oil, 30 lbs.; pale tar oil, 10 lbs.; paraffin oil, 80 lbs.; heavy spar, 80 lbs.

Bring the lime meal into an iron kettle, add gradually the blue rosin oil and stir until saponification takes place. Then add, with vigorous stirring, the pale tar oil and, in case saponification is incomplete, stir in ½ to ¾ lb. of caustic lye of 5° Bé. If necessary, warm the mass and finally add slowly, with constant stirring, the paraffin oil as well as the heavy spar.

For coloring this grease black-brown lamp-black is used; for violet, Frankfort black; for blue, Berlin blue.

*In the warm way.* I. Heavy paraffin oil, 60 lbs.; rosin oil, 60 lbs.; tallow, 60 lbs.; oil foots, 30 lbs.; finely pulverized burnt lime, 15 lbs.; soda lye of 40° Bé., 6 lbs.

Melt with the assistance of heat in strong iron kettle the paraffin oil, rosin oil and oil foots, and saponify the mixture with the lime and soda lye.

II. Palm oil, 50 lbs.; tallow, 20 lbs., soda lye, 15 lbs.; boiling soft water, 16½ lbs.; cold soft water. 200 lbs.

Melt the palm oil and tallow in an iron kettle and gradually add the soda lye and, as the mass becomes thick, pour in the above-mentioned quantity of hot water, stirring constantly. The mixture is allowed to stand in the air for 50 to 60 hours and then thoroughly pressed in a cooling vat whereby, with constant stirring, the above-mentioned quantity of cold water is added.

III. Fresh fat lime, 600 lbs.; paraffin oil, 1000 lbs.; tar oil, 600 lbs.; rosin oil, 1000 lbs.

The lime is gradually slaked to a thick paste and the latter brought into an iron kettle. While the paste is still boiling hot, the paraffin oil is added with vigorous stirring. At 68° to 75° F. an intimate combination of the lime with the oils takes place, all particles of water being completely separated. The water is removed from the thin paste and the latter allowed to cool to 64° F., when the tar oil and finally the rosin oil are added. If the grease is to be filled stir into the cold mass 50 lbs. of talc for every 200 lbs. of oil.

IV. *Yellow Belgian axle grease.* Crude rosin oil, 525 lbs.; lime slaked to powder, 400 lbs.; yellow paraffin oil, 250 lbs.

Boil in a capacious strong iron kettle over a moderate fire, with constant stirring, the lime, with 500 lbs. of crude rosin oil until every particle of water has disappeared and the mass has become homogeneous. Now remove the fire and stir the viscous mass until it is so stiff that it can no longer be moved. This product is the "basis-mass."

To prepare from it the yellow axle grease, briug 250 lbs. of yellow paraffin oil into an iron kettle, add, with constant stirring, 100 lbs. of the basis-mass, and stir in 25 lbs. of crude rosin oil to give the grease the proper consistency.

### *Manufacture of English Patent Axle Grease.*

*Preparation of the basis-mass.* Train oil, 350 lbs.; slaked lime, 249 lbs.

Bring the train oil into a capacious iron boiler of about 100 gallons capacity, and boil so long as water evaporates, which requires about 25 to 35 minutes. Then stir in the slaked lime previously divided into three equal portions. The first portion of the lime must be introduced slowly and carefully, otherwise the contents of the kettle run over. The two remaining portions of the lime are then gradually added with constant stirring and the whole kept boiling until it acquires a viscous consistency. Care must be taken frequently to stir the contens of the kettle to prevent any of the lime from settling on the bottom. About an hour after the introduction of the slaked lime the mass is thoroughly worked through and then brought into a low, open vat to cool.

*Blue patent axle grease, also called paraffin fat.* Basis-mass, 100 lbs.; rosin oil, 225 lbs.; coal tar oil, 25 lbs.; wood tar oil, 3½ lbs.

Bring the thoroughly cooled rosin oil into a large vat, add the coal tar oil and wood tar oil, stir thoroughly, and add the basis-mass with constant vigorous stirring until the mass thickens.

This axle grease has a dark blue color, and is of the consistency of butter.

*White patent axle grease. White basis-mass.* Soft water, 200 lbs.; calcined soda, 25 lbs.; finely sifted slaked lime, 20 lbs.; rosin oil, 150 lbs.

Bring the water into an iron kettle, dissolve therein, by boiling, the calcined soda, and when solution is complete add the lime, continuing the boiling until a homogeneous mass has been formed. Finally add the rosin oil and boil for 15 minutes, stirring the mixture at intervals.

The product thus obtained forms the basis mass for white grease. It is recommended not to use this basis mass at once, but to let it stand till the next day, the quality of the axle grease being the better the colder the basis mass is.

*Preparation of white patent axle-grease.* White basis-mass, 200 lbs.; rosin oil, 225 lbs.; basis-mass, prepared with rosin and tar oils, 100 lbs.

Bring the white basis-mass into a vat, add the rosin oil, and stir the mixture until the whole has combined to a white mass of the consistency of syrup, which as a rule requires about three minutes. Now add the basis-mass prepared with rosin and tar oils, as used for blue axle-grease, and stir vigorously till the mass thickens.

*Black patent axle-grease.* White basis-mass, 125 lbs.; lamp-black, 8 oz.; wood tar oil, 25 lbs.; coal tar oil, 37½ lbs.; heavy rosin oil, 150 lbs.; light rosin oil, 25 lbs.; basis-mass of blue patent axle-grease, 100 lbs.

Bring the white basis-mass into a vat, and add the lamp-black and the wood tar and coal tar oils. Vigorously triturate the lamp black with the basis-mass and oils until the mass has acquired a dark color. Now add the heavy, and finally the light, rosin oil. Stir the whole thoroughly and whilst doing so gradually add the basis-mass of blue patent axle grease.

This axle grease has a blue color and a beautiful lustre.

*Violet patent axle grease.* White basis-mass, 125 lbs.; Frankfort black, 8 ozs.; wood tar oil, 25 lbs.; coal tar oil, 37½ lbs.; heavy rosin oil, 150 lbs.; light rosin oil, 25 lbs.; basis-mass of blue patent axle grease, 100 lbs. Prepared in the same manner as black patent axle grease.

*Ordinary English axle grease.* I. Purified tallow, 120 lbs.; palm oil, 240 lbs.; water, 160 lbs.; soda, 20 lbs.

II. Purified tallow, 160 lbs.; palm oil, 400 lbs.; water, 200 lbs.: soda, 30 lbs.

Melt the tallow in an iron kettle, then heat it to 248° F., and stir in the palm oil. Dissolve the soda by means of warm water, and, with constant stirring, add the solution in a thin stream to the contents of the kettle. When the last portion of the soda solution has been added, remove the kettle from the fire, and stir until the contents commence to solidify.

By using more tallow, this axle grease becomes more solid, while, on the other hand, by using more palm oil it melts at a lower temperature and acquires a more butter-like consistency.

III. Palm oil, 84 lbs.; tallow, 34 lbs.; soda lye, 26 lbs.; water, 268 lbs.

Melt the palm oil together with the tallow in an iron kettle, effecting an intimate combination by vigorous stirring. Now add, with constant stirring, the above mentioned quantity of soda lye of 20° Bé., which is prepared by dissolving soda in hot water until the cooled liquid shows 20° Bé. Next pour the water into the kettle, and stir until the mass is perfectly homogeneous, when it is brought into vessels to solidify.

IV. Palm oil, 80 lbs.; train oil, 400 lbs.; caoutchouc, 8 lbs.; litharge 8 lbs.; lead acetate (sugar of lead), 8 lbs.

Comminute the caoutchouc and heat it together with the train oil in an iron kettle to 383° or 392° F. Filter the contents of the kettle, add to the filtrate the litharge and acetate of lead, return the whole to the kettle, and heat for one hour at 392° F. Then add with vigorous stirring, the palm oil.

V. Tallow, 150 lbs.; lard, 37½ lbs.; palm oil, 37½ lbs.; graphite, 9 lbs.

This so called *graphite axle grease* is prepared by intimately mixing together the ingredients by passing steam through the vat containing them.

VI. Paraffin oil, 60 lbs.; purified colza oil, 440 lbs.

Heat the paraffin oil and colza oil, each by itself, then mix them and vigorously stir the mixture for some time. The above quantities suffice for summer use; for winter use take: paraffin oil, 36 lbs.; purified colza oil, 564 lbs.

VII. Asphalt, 100 lbs.; black pitch, 25 lbs.; petroleum, 25 lbs.; litharge, 25 lbs.; water, 250 lbs.

Melt in an iron kettle the asphalt together with the pitch, and then add carefully the petroleum in a thin jet until the contents of the kettle are uniformly liquid. Then bring the litharge into the kettle and add gradually the water, stirring the mass vigorously until it is perfectly homogeneous. By using more or less petroleum the degree of fluidity of this axle grease may be increased or reduced.

VIII. Asphalt, 80 lbs.; pitch, 20 lbs.; litharge, 20 lbs.; water, 160 to 200 lbs.

Melt the pitch together with the asphalt in an iron kettle over a moderate fire, add the litharge together with the water, heat the whole with vigorous stirring to boiling, and when the contents of the kettle have been converted into a fatty mass, remove the grease from the fire.

*English axle grease specialties. Bluish axle grease.* Unslaked lime, 150 lbs.; rectified heavy tar oil, 250 lbs.; rosin oils, oil foots, or paraffin oil, 250 to 300 lbs.

Slake the lime with water in a strong iron kettle, then stir in gradually enough water to form a thin lime paste, and pour the tar oil into the boiling mass. By continuously stirring the mass the lime enters into an intimate combination with the oils at 122° F., the water being completely separated and is carefully drawn off through a cock in the bottom of the kettle. When the temperature is reduced to about 80° F., the rosin oil, or one of the above-mentioned substitutes for it, is stirred into the mass.

The product obtained is of the consistency of butter, and has a bluish color.

*Blue axle grease.* Finely elutriated graphite, 3 lbs.; ultra-marine, 3 lbs.; soft soap, 6 lbs.; lard, 24 lbs.; colza oil, 3 lbs.

Into a suitable vat, provided with stirring apparatus, bring the graphite, ultramarine, soft soap and lard, and combine them by stirring. When combination has been effected, add the colza oil, and continue stirring until a homogeneous mass is formed.

*Yellow axle grease.* Red American rosin, 100 lbs.; caustic soda lye of 20° Bé., 12½ lbs.; crude rosin oil, 12½ lbs.

Melt the rosin in an iron kettle, add gradually the lye, and then, with vigorous stirring, the rosin oil.

*Black axle grease.* Finely elutriated graphite, 9 lbs.; soft soap, 6 lbs.; lard, 36 lbs.

In a suitable vessel incorporate the graphite with the soft soap and lard by vigorous stirring.

*Gray-black axle grease.* Lard, 50 lbs.; finely elutriated graphite, 10 lbs.

Melt the lard in an iron kettle over a moderate fire, and stir the graphite into the melted mass.

II. Spermaceti, 20 lbs.; yellow wax, 10 lbs.; mutton suet, 40 lbs.: lard, 10 lbs.; finely elutriated graphite, ½ lb.

Melt the spermaceti, yellow wax, suet and lard in an iron kettle, and while the mass is cooling stir in the graphite.

*English palm oil axle grease.* Red American rosin, 100 lbs.; palm oil, 6½ lbs.; rectified rosin oil, 18½ lbs.; caustic soda lye of 22° Bé. 24½ lbs.; water, 2 lbs.

Melt the rosin in an iron kettle over a moderate fire, then, in order to saponify the rosin, add the lye and the water. The melted palm oil and the rosin oil are next brought into the kettle and intimately mixed with the saponified rosin, stirring being continued without interruption, until a homogeneous product is obtained.

### *Fluid Lubricants.*

Fluid lubricants possess many advantages over the solid ones, and are frequently preferred by railroad men and manufacturers of machinery. The principal advantage is that they do not require complicated oiling arrangements (oil-cups, etc.], and that they act without previous friction being required to give to the lubricant the requisite fluidity. They have further the valuable quality that their consistency does not so much depend on the temperature of the air as is the case with solid lubricants.

The principal materials used in the manufacture of fluid lubricants on a large scale, and which have stood the test of time, are: 1. Rape-seed and colza oils. 2. Olive oil. 3. Rosin oil, either by itself or in combination with lime and certain products of dry distillation (paraffine). 4. Train oil. 5. Neat's foot oil and bone oil. 6. The so-called mineral oils. 7. Petroleum and ozokerite. 8. Solutions of soap.

The oils as obtained by pressure and other means are not directly available for lubricating purposes, but have to be purified. This purification or refining is effected by various processes described in Vol. 1, Chapter XIII., page 366.

### *Cohesion Oils.*

It cannot be denied that many lubricants, and especially fluid ones, are consumed very rapidly, so that where a great number of axles, for instance upon railroads, are in continuous activity, the expenses run very high, though the lubricant itself is all that can be desired.

To overcome this evil and to reduce the consumption of lubricant to a minimum, the so-called cohesion oils have been introduced, and it must be said they answer the purpose, as the quantity consumed is actually much less than is the case with other lubricants.

As indicated by the name, cohesion oils are less fluid than ordinary fluid lubricants. The opinions about their usefulness vary very much. While some manufacturers who use large quantities of lubricants are thoroughly satisfied with them and consider them superior to refined rape-seed oil or olive oil, others complain that by depositing a sediment they contaminate the journals and oiling arrangements to such an extent as to stop all regular lubrication.

A cohesion oil which shows the last-mentioned properties belongs, according to our opinion, to the worst products. In order to perfect their product many manufacturers forced cohesion too far and finally brought into commerce oils running sluggishly from the can, and forming threads like congealing glue. Such an oil containing an excess of substances to render it more thickly fluid, would of course deposit a portion of these substances and form a thickly fluid mass not adapted for lubrication.

The composition of cohesion oils is, as a rule, kept a secret, the manufacturer frequently adding coloring and scenting substances, entirely indifferent as lubricants, for the purpose of disguising the nature of the other ingredients and rendering their recognition more difficult. But the skilled chemist can nevertheless analyze these products, and many examinations of American, English, and German cohesion oils show that the principal materials entering into their composition are nearly the same in all of them.

The basis of cohesion oils is, without exception, a more or less thickly fluid fat, most frequently crude rape-seed oil; train oil is less frequently used, and then is generally mixed with tallow, palm oil, or some other solid fat to increase the degree of fluidity.

Besides fat, cohesion oils contain varying quantities of rosin oil, from 8 to 20 per cent. of the quantity of fat used.

Common American rosin is used as an addition to impart to the cohesion oils their peculiar characteristic consistency; the greater the quantity added the more the cohesion of the lubricant is increased. Generally 8 to 15 per cent. of the quantity of fat is used. It is not advisable to exceed this, and especially when the lubricant is to be used at a low temperature.

The preparation of cohesion oil is quite simple. Heat the rape-seed oil moderately in a boiler, and, if necessary, add the required quantity of solid fats (tallow, palm oil, etc.). In a second boiler, specially protected against the ignition of its contents, heat the rosin oil nearly to the boiling-point and add the comminuted rosin in small portions, allowing one portion to dissolve entirely before putting in the next. During this operation the contents must be constantly stirred to prevent the rosin from settling on the bottom and scorching.

When all the rosin is dissolved the solution of rosin and rosin oil is ladled into the boiler containing the oil, with constant stirring. The fire under the oil-boiler is then withdrawn, but the stirring continued until the mass commences to acquire a thickly fluid consistency.

In the following we give the composition of two cohesion oils, of which *No.* 1 is the most thickly fluid and calculated for quick-running axles carrying heavy loads, and *No.* 2 for light axles.

Other circumstances being equal, *No.* 1 may be used in summer and *No.* 2 in winter.

*Cohesion oil.—No.* 1. Crude rape seed oil, 95 parts; purified tallow, 5; rosin oil, 12; American rosin, 12.

*No.* 2. Crude rape-seed oil, 96 parts; purified tallow, 4; rosin oil, 8; American rosin, 8.

### *Train-oil Lubricants.*

In this country the value of train oil for lubricating purposes is more highly appreciated than in Europe.

To convert crude train oil, which generally is brown and of a bad odor, into a good lubricant, it is best to expose it to a low temperature, whereby a large portion of the solid fat is separated, and then to treat it with litharge to fix the free oleic acid which may be present.

The litharge is to be ground as fine as possible, and triturated with some train oil, so that a thick viscid mass is formed. This is diluted with sufficient train oil to make it thinly fluid, and is then mixed with the remainder of the train oil by thorough stirring. The mass is then allowed to stand quietly for some time, whereby the excess of litharge used is deposited on the bottom, while the newly-formed combination of oleic acid and litharge remains dissolved in the train oil, making it more thickly fluid.

### *Lubricants for Special Purposes.*

*Lubricants for axles running at high speed:* No. 1. Soap, 1 part; colza oil, 1; water, 5; talc powder, 2.

No. 2. Brown ozokerite, 10 parts, petroleum, 4.

To prepare No. 1, the constituents are boiled and stirred; for No. 2, melting together of the ozokerite and petroleum suffices.

*Lubricant for piston rods.* Paraffine, 1 part, and talc powder, 4 parts, are stirred together while hot. Wicks are dipped in the mixture, and pressed into the stuffing-boxes of the piston rods. One lubrication with this preparation suffices for eight to fourteen days.

*Lubricant for cog-wheels.* Any butyaceous lubricant is melted and mixed with 5 per cent. of its weight of finely-ground glass. After a short use this lubricant makes the cogs perfectly smooth and even.

*Lubricants for belts.* Linseed oil, 45 parts; litharge, 20; water, 20. Boil the three constituents until the mass has acquired the consistency of plaster, and, while hot, add sufficient oil of turpentine to give it the consistency of varnish.

*Belgian wagon grease.* Lime slaked to a powder, 100 parts; tar oil, 300; paraffine oil, 800; rosin oil, 300; strong lye, 12.

Bring the slaked lime powder and the lye into a kettle, add the rosin oil, and stir until the entire mass is white. Then heat slightly, add with constant stirring the tar oil, then the paraffine oil, and, finally, 800 parts of talc powder. The compound is then stirred until it is entirely homogeneous.

*Coll's patent lubricant* consists entirely of rosin oil boiled with a quantity of slaked lime, not sufficient for saponification, and is, therefore, actually a rosin oil-lime soap mixed with an excess of rosin oil. Being entirely free from acid it can be recommended as a lubricant.

*Lubricating oils for watchmakers and for sewing machines.* Lubricating oils intended for watches or other delicate machinery are chiefly prepared from refined colza oil, or, still better, from fine olive oil.[*] To free the oils from the last traces of adhering acid shake them vigorously and repeatedly with one-hundredth part of their weight of caustic-soda lye. After allowing the oil to remain for several days in contact with the caustic lye, add a large quantity of water, and carefully pour off the oil floating on top, which is now entirely free from acid.

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[*] For watch oil from porpoise and blackfish oils, see Vol. 2, p. 48.

The oil contains, however, coloring matter and other substances exerting an injurious effect. These are removed by means of strong alcohol in the following manner: Bring into a bottle of white glass of about 13 quarts' capacity, 10 quarts of the oil to be purified, pour in 2 quuarts of very strong alcohol (90 per cent.), close the bottle with a well-fitting cork, and mix the oil and alcohol by shaking. Place the bottle in the sun, and repeat the shaking several times a day.

In the course of three weeks—in summer frequently in ten to fourteen days—the oil will have become clear as water, and floats upon the alcohol, which has withdrawn the coloring substance from the oil, and acquired a yellow color.

The purified oil is drawn off by means of a siphon into small bottles, which are tightly corked and kept in a dark, cool place. By careful distillation the alcohol can be freed from the absorbed coloring matter, so that it can be re-used for the treatment of fresh quantities of oil.

According to special experiments, fine olive oil or fresh expressed sweet-almond oil bleached in the above manner is best adapted, either each by itself or mixed in equal parts for lubricating watches.

*Fat watch oil.* Watchmakers and manufacturers of mechanical instruments are generally willing to pay a high price for a lubricating oil answering their requirements. Such a lubricant must exert no effect upon the metals nor become thickly fluid, nor resinify in the course of time.

Many experiments made in this direction show that there are actually but two substances—olive oil and pure bone oil—suitable for the purpose.

*Olive-oil lubricant for watches.* Refine the olive oil with sulphuric acid, and after careful washing, shake the oil with about 2 per cent. of weak lye, to remove the last traces of adhering acid, and let it stand for a few days. Then draw off the oil floating upon the surface, and treat it with alcohol in the manner previously described.

Like every other fine lubricating oil, the olive oil is kept in small well-corked bottles.

*Bone-oil lubricant for watches.* Bone oil is especially adapted for the lubrication of watches and other fine machinery. For tower-clocks it is especially valuable, as it possesses the property of remaining entirely fluid at a low temperature, while most other oils become entirely solid or at least very thickly fluid, at a few degrees below the freezing point of water.

To purify the oil for this purpose, expose thoroughly refined oil to a temperature near the freezing point of water. By keeping it at this temperature for several hours a solid mass of fat is separated, which is removed from the portion remaining liquid. This manner of purification is readily accomplished in winter. To be able to do it in summer, place the vessel containing the bone oil in a vessel with cold water and keep the temperature near the freezing-point by adding ice.

A finer product, constituting in fact the best lubricant, is obtained by treating the bone oil in the following manner: Fill a bottle one-third full with the oil to be purified. Then pour benzine clear as water in small portions upon the oil, close the bottle and shake until the benzine has disappeared. By again adding benzine and shaking, a complete solution of the fat is finally effected. That this has actually taken place is recognized by the contents of the bottle not separating into two layers after long standing.

The bottle is then exposed to a low temperature for several hours. A solid fat deposits on the bottom, and the lower the temperature the greater the deposit. Alongside the bottle containing the oil, place another bottle with a funnel, the lower end of which is closed by a cotton stopper. After thoroughly shaking the bottle with oil, pour the contents into the funnel; the fluid portion runs into the bottle, while the solid portion is retained in the funnel by the cotton stopper. The clear solution of bone oil in benzine collected in the bottle is then brought into a small retort, which is connected with a thoroughly cooled receiver. Place the retort in a tin vessel filled with water and apply heat. The benzine readily distils off, leaving the purified bone oil in the retort. The benzine condensed in the receiver can be re-used for the solution of fresh portions of bone oil.

For distilling off the benzine the apparatus (Fig. 269) described on Vol. 2, page 302, can be advantageously used.

After allowing all the benzine solution to drain from the funnel, the latter is placed upon another bottle and the whole removed into a warm room. The solid mass separated in the cold soon melts and filters through the cotton. It is an excellent lubricant for fine machines not exposed to a low temperature.

*Mineral oil for watchmakers.* This is a heavy tar oil, which is purified in a peculiar manner. Compound 100 parts of ordinary heavy tar oil with 2 parts of calcium chloride, stirring it thoroughly into the oil, and add 3 parts of crude hydrochloric acid. Stir thoroughly and let the mass rest for six hours. Then pour off the oil and shake it repeatedly with 5 parts of caustic soda. Finally filter the purified oil through gray blotting-paper.

*Caoutchouc lubricant for sewing-machines.* Olive oil, almond oil, colza oil, each 50 parts; caoutchouc, 2; carbon disulphide, 4.

Dissolve the caoutchouc in the carbon disulphide in a well-closed bottle, filter the solution quickly into a vessel containing the oil, place the latter in a vessel containing water, and apply heat until the water boils, stirring the mixture constantly.

Care should be had not to inhale the escaping noxious vapors of carbon disulphide and have no fire or light in the room, the vapors being very inflammable and a mixture of them with air causing explosion.

*Mineral oils for sewing-machines.* Petroleum, 100 parts: water, 10: calcium chloride, 1. Dissolve the calcium chloride in water, combine the filtered, clear solutions with the petroleum and shake frequently. After two hours separate the oil from the calcium chloride solution and shake it with a solution of caustic potash in water. Finally bring the oil, together with the caustic potash solution, into still and distill it over.

*Sewing machine and watchmaker's oil.* Treat a mixture of olive oil, 3 parts; almond oil, 2; and colza oil, 1; with alcohol in the manner mentioned on page 666. This lubricant is quite thinly fluid and is suitable for lubricating very delicate portions of machines.

# LIST OF PATENTS RELATING TO FATS AND OILS ISSUED BY THE GOVERNMENT OF THE UNITED STATES OF AMERICA, FROM 1790 TO 1896, INCLUSIVE.*

## *Extraction of Fats and Oils.*

Homer T. Yaryan shows complex arrangements of coils and tanks which have been successful; Adamson has the use of hydrocarbon solvents by circulation from vat to vat through the material to be treated; and Byerly a revolving vacuum filter.

The following patents relate to the subjects specified: Menthol, 330,274, and reissue 10,705; hotel grease, 156,815; paraffine oil, 346,448; enfleurage by petroleum, 243,510; essential oils, 120,596; steaming seed and meal, 334 and 308,112; moistening seeds, 163,933, 168,164, 202,839; maize oil, 313,665; cod-liver oil, 275,828 and 353,090; suint, 71,191 and reissue, 3,239; from leather stock, 300,497 and 332,840; use of cork mats, 328,925; for removing oil from castings, 389,058; for getting oil from garbage, 425,603; for cottonseed oil, 428,134; for cod-liver oil, 459,873; for removing ink and grease from plate printers' gauze, 471,323; for getting oil from garbage, 516,769; for fish oil, 520,242; for getting oil from garbage, 532,799 and 533,940; for cottonseed oil, 545,900 and 550,628.

| No. | Date. | Inventor. | Residence. |
|---|---|---|---|
| 334 | July 31, 1837. | James Criswell, | Pittsburgh, Pa. |
| 4,961 | Feb. 9, 1847. | E. Horner, | East Brook, Pa. |
| 20,048 | April 27, 1858. | E. Deiss, | Paris, France. |
| 26,845 | Jan. 17, 1860. | G. G. Henry, | Mobile, Ala. |
| 45,584 | Dec. 27, 1864. | J. Chilcott, | Brooklyn, N. Y. |
| 46,033 | Jan. 24, 1865. | L. Smith, | Erie, Pa. |
| 46,809 | Mar. 14, 1865. | B. G. Martin, | Philadelphia, Pa. |
| 46,945 | Mar. 21, 1865. | T. Rowe, | Brooklyn, N. Y. |
| 48,107 | June 6, 1865. | L. Smith, | Erie, Pa. |
| 50,626 | Oct. 21, 1865. | S. W. Pingree, | Lawrence, Mass. |
| 55,915 | June 26, 1866. | J. Schneider, | Williamsburg, N. Y. |
| 61,315 | Jan. 22, 1867. | D. Bruce, | Rossville, N. Y. |
| 61,463 | Jan. 22, 1867. | J. Robertson, | Brooklyn, N. Y. |
| 66,119 | June 25, 1867. | J. Besso, | Philadelphia, Pa. |
| 68,506 | Sept. 3, 1867. | C. O. Heyl, | Berlin, Prussia. |
| 71,191 | Nov. 19, 1867. | O. Macdaniel, | New York, N. Y. |
| Reissue 3,239 | Dec. 22, 1868. | " | " |

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* The American patent system was founded by Act of April 10, 1790. After the fire, which destroyed the Patent Office in 1836, the Government advertised for the patents which had been issued prior to that time. In this way numerous copies of the earlier patents were secured. This explanation seems necessary in view of the incompleteness in the earlier dates of these lists.

| No. | Date. | Inventor. | Residence. |
|---|---|---|---|
| 88,222 | Mar. 23, 1860. | Sim and Hutchinson, | Baltimore, Md. |
| 93,645 | Aug. 10, 1869. | T. Simm, | " |
| 105,688 | July 26, 1870. | E. S. Hutchinson, | " |
| 110,998 | Jan. 17, 1871. | G. G. Percival, | Waterville, Maine. |
| 111,064 | Jan. 17, 1871. | E. S. Hutchinson, | Baltimore, Md. |
| 111,658 | Feb. 7, 1871. | W. W. Lucas, | Cleveland, O. |
| 112,339 | Mar. 7, 1871. | T. Sim, | Baltimore, Md. |
| 112,350 | Mar. 7, 1871. | E. S. Hutchinson, | " |
| 112,628 | Mar. 14, 1871. | J. J. Powers, | Memphis, Tenn. |
| 117,135 | July 18, 1871. | W. Adamson, | Philadelphia, Pa. |
| 117,136 | July 18, 1871. | " | " |
| 118,668 | Sept. 5, 1871. | " | " |
| 119,187 | Sept. 19, 1871. | C. F. A. Simonin, | Philadelphia, Pa. |
| 119,188 | Sept. 19, 1871. | " | " |
| Reissue 4,578 | Oct. 3, 1871. | L. S. Robbins, | New York, N. Y. |
| 120,596 | Nov. 7, 1871. | G. G. Percival, | Waterville, Maine. |
| 126,300 | April 2. 1872. | E. S. Hutchinson, | Baltimore, Md. |
| 128,594 | July 2, 1872. | A. N. Cole, | Brockville, Canada. |
| 146,339 | Jan. 13, 1874. | L. Herreshoff, | Bristol, R. I. |
| 151,050 | May 19, 1874. | G. N. Phelps, | Brooklyn, N. Y. |
| 156,751 | Nov. 10, 1874. | J. C. Bates, | Providence, R. I. |
| 156,815 | Nov. 10, 1874. | H. Pindar, | New York, N. Y. |
| 160,550 | Mar. 9, 1875. | R. Scaife, | London, Eng. |
| 163,130 | May 11, 1875. | W. Adamson, | Philadelphia, Pa. |
| 163,933 | June 1, 1875. | A. B. Lawther, | Chicago, Ill. |
| 168,934 | Sept. 28, 1875. | " | " |
| 170,424 | Nov. 23, 1875. | J. R. Whitney and W. Lawrence. | New York, N. Y. |
| 174,761 | Mar. 14, 1876. | G. Bent, | Lowell, Mass. |
| 176,851 | May 2, 1876. | W. M. Force, | Newark, N. J. |
| 183,097 | Oct. 10, 1876. | W. Adamson, | Philadelphia, Pa. |
| 183,098 | Oct. 10, 1876. | " | " |
| 184,815 | Nov. 28, 1876. | " | " |
| 191,635 | June 5, 1877. | " | " |
| 202,839 | April 23, 1878. | A. B. Lawther, | Chicago, Ill. |
| 203,980 | May 25, 1878. | W. Adamson and C. F. A. Simonin, | Philadelphia, Pa. |
| 203,981 | May 25, 1878. | | |
| 205,328 | Jnne 25, 1878. | Homer T. Yaryan, | Richmond, Ind. |
| 205,516 | July 2, 1878. | " " | " |
| 212,878 | Mar. 4, 1879. | W. Adamson, | Philadelphia, Pa. |
| 220,203 | Sept. 30, 1879. | Homer T. Yaryan, | " |
| 224,075 | Feb. 3, 1880. | S. G. Clark, | Cleveland, O. |
| 238,543 | Mar. 8, 1881. | Homer T. Yaryan, | Richmond, Ind. |
| 241,772 | May 17, 1881. | " " | Toledo, O. |
| 243,510 | June 28, 1881. | R. A. Chesebrough, | New York, N. Y. |
| 245,294 | Aug. 9, 1881. | F. X. Byerly, | Cleveland, O. |
| 245,365 | Sept. 9, 1881. | J. W. Evans, | " |
| 245,765 | Aug. 16, 1881. | C. T. Basland, | London, Eng. |
| 246,075 | Aug. 23, 1881. | F. X. Byerly, | Cleveland, O. |
| 246,172 | Aug. 23, 1881. | W. M. Matthews and M. Lancaster, | Richmond, Ind. |
| 248,353 | Oct. 18, 1881. | R. S. Perrin, | Yew York, N. Y. |
| 253,378 | Feb. 7, 1882. | E. B. Hart, | Strandtown, Belfast, Ire. |
| 253,379 | Feb. 7, 1882. | " | " " |
| 253,722 | Feb. 14, 1882. | M. Lancaster, | Richmond, Ind. |
| 260,182 | June 27, 1882. | J. B. Field, | Ottumwa, Ia. |
| 261,634 | July, 25, 1882. | F. Seltsam, | Forcheim, Bavaria. |

| No. | Date. | Inventor. | Residence. |
|---|---|---|---|
| 265,517 | Oct. 3, 1882. | M. Lancaster, | Richmond, Ind. |
| 270,291 | Jan. 9, 1883. | S. B. Dour, | Dayton, O. |
| 271,664 | Feb. 6, 1883. | C. T. Stollmeyer, | Trinidad, W. I. |
| 271,859 | Feb. 6, 1883. | J. D. Jones, | Brooklyn, N. Y. |
| 275,642 | April 10, 1883. | C. B. Gordon, | Grand Crossing, Ill. |
| 275,828 | April 17, 1883. | A. Kenrick and E. L. Jerauld, | Harwich, Mass. |
| 275,989 | April 17, 1883. | F. X. Byerly, | Cleveland, O. |
| 280,483 | July 3, 1883. | E. Kells and A. J. Adamson, | " |
| 280,549 | July 3, 1883. | H. T. Yaryan, | Richmond, Ind. |
| 280,550 | July 3, 1883. | " | " |
| 281,039 | July 10, 1883. | J. W. Evans, | Cleveland, O. |
| 281,040 | July 10, 1883. | " | " |
| 281,566 | July 17, 1883. | Sam'l and Adolph Scheibler, | Memphis, Tenn. |
| 285,220 | Sept. 18, 1883. | R. B. Brown, | St. Louis, Mo. |
| 285,598 | Sept. 25, 1883. | J. W. Evans, | Cleveland, O. |
| 286,553 | Aug. 29, 1883. | J. T. O'Shaughnessy, | New York, N. Y. |
| 287,382 | Oct. 23, 1883. | J. D. Jones, | Brooklyn, N. Y. |
| 288,259 | Nov. 13, 1883. | G. N. Phelps, | " |
| 290,083 | Dec. 11, 1883. | A. P. Massey, | Cleveland, O. |
| 294,425 | Mar. 4, 1884. | A. J. Adams, | " |
| 296,187 | April 1, 1884. | W. Krutzsch, | Dayton, O. |
| 297,743 | April 29, 1884. | J. A. Bang and A. Sanguineth, | Marseilles, France. |
| 298,816 | May 20, 1884. | M. A. Beall, | Chicago, Ill. |
| 300,497 | June 17, 1884. | T. T. Newell, | Chelsea, Mass. |
| 305,224 | Sept. 16, 1884. | N. A. Pratt and G. W. Benson, | Marietta, Ga. |
| 306,653 | Oct. 14, 1884. | R. H. Smith, | Carbondale, Pa. |
| 306,733 | Oct. 21, 1884. | L. H. Friedburg, | New York, N. Y. |
| 306,734 | Oct. 21, 1884. | " | " |
| 306,735 | Oct. 21, 1884. | " | " |
| 307,123 | Nov. 4, 1884. | G. Leder, | Demopolis, Ala. |
| 307,124 | Nov. 4, 1884. | " | " |
| 307,707 | Nov. 4, 1884. | F. X. Byerly, | Cleveland, O. |
| 308,112 | Nov. 18, 1884. | J. H. Vaile, | Dayton, O. |
| 308,436 | Nov. 25, 1884. | F. Seltsam, | Forcheim, Bavaria,Ger. |
| 309,312 | Feb. 16, 1885. | G. Patry, | Paris, France. |
| 312,674 | Feb. 24. 1885. | A. E. Thornton, | Atlanta, Ga. |
| 313,440 | Mar. 3, 1885. | J. L. Moss and N. Littell, | Rome, Ga. |
| 313,665 | Mar. 10, 1885. | F. V. Greene, | Philadelphia, Pa. |
| 318,191 | Mar. 19, 1885. | G. Leder, | Demopolis, Ala. |
| 322,425 | July 21, 1885. | G. O. Cromwell and H. E. Coy, | Toledo, O. |
| 328,925 | Oct. 27, 1885. | J. Davis, | Charleston, S. C. |
| 330,274 | Nov. 10, 1885. | A. M. Todd, | Nottawa, Mich. |
| 331,895 | Nov. 8, 1885. | J. A. Lewis, | Meridian, Miss. |
| 332,842 | Dec. 22, 1885. | J. I. Sawin, | Worcester, Mass. |
| 336,175 | Feb. 16, 1886. | D. A. Tompkins and F. Oliver, | Charlotte, N. C. |
| Reissue 10,705 | Mar. 30, 1886. | A. M. Todd, | Nottawa, Mich. |
| 346,448 | July 27, 1886. | C. Vose, | Brooklyn, N. Y. |
| 353,090 | Nov. 23, 1886. | J. W. Stairs, and J. Craig, | Halifax, N. S. |

| No. | Date. | Inventor. | Residence. |
|---|---|---|---|
| 353,823 | Dec. 7, 1886. | C. A. Sahlstrom, | Jonkoping, Sweden. |
| 354,299 | Dec. 14, 1886. | C. Baumgarten, | Schulenberg, Tex. |
| 360,996 | April 12, 1887. | W. Buttner, | Cummersbach, Prussia. |
| 369,357 | Sept. 13, 1887. | R. Hunt and E. S. Wilson, | Liverpool, England. |
| 379,808 | Mar. 20, 1888. | F. A. Owen and J. A. Harvey, | Burlington, Vt. |
| 381,042 | April 10, 1888. | J. W. Evans, | Cleveland, O. |
| 389,058 | July 4, 1888. | E. L. Cole, | Chicago, Ill. |
| 393,023 | Nov. 20, 1888. | N. C. Nagel, R. H. Kaemp, A. W. F. G. Linnenbrugge, | Hamburg, Germany. |
| 393,972 | Dec. 4, 1888. | E. T. Gennert, | N. Y. City. |
| 393,973 | Dec. 4, 1888. | “ | “ |
| 397,792 | Feb. 12, 1889. | W. T. Forbes, | Atlanta, Ga. |
| | | G. Leder, | Demopolis, Ala. |
| 406,781 | July 9, 1889. | J. Singer and M. W. Judell, | Adelaide, S. Australia. |
| 410,291 | Sept. 3, 1889. | H. Pindar, | N. Y. City, |
| 421,154 | Feb. 18, 1890. | J. H. Vaile, | Dayton, O. |
| | | D. A. Tompkins, | Charlotte, N. C. |
| 425,603 | April 15, 1890. | G. Fleischman, | Buffalo, N. Y. |
| 427,410 | May 6, 1890. | W. T. Forbes, | Atlanta, Ga. |
| 428,134 | May 20, 1890. | J. S. Price, | Houston, Tex. |
| 431,937 | July 8, 1890. | H. A. Dombrain, | Leeds, Yorkshire, Eng. |
| | | O. T. Engle, | Stratford, Essex, Eng. |
| 432,808 | July 22, 1890. | P. C. Vogellus, | Philadelphia, Pa. |
| 433,152 | July 29, 1890. | M. Schroeder, | Neumuhl, Hamborn, Germany. |
| 434,696 | Aug. 19, 1890. | C. F. Binder, | Edgewood, Ga. |
| 436,227 | Sept. 9, 1890. | W. T. Forbes, | Atlanta, Ga. |
| 436,623 | Sept. 16, 1890. | G. S. Andrews, | San Francisco, Cal. |
| 436,624 | | | |
| 440,418 | Nov. 11, 1890. | G. and A. Burnell, | Hindmarsh, Australia, |
| 440,963 | Nov. 18, 1890. | L. Pinagel, | Aix la Chapelle, Ger. |
| 442,081 | Dec. 2, 1890. | O. R. Nelson, | Chicago, Ill. |
| 443,407 | Dec. 30, 1890. | J. W. Evans, | Cleveland, O. |
| 449,215 | Mar. 31, 1891. | “ | “ |
| 451,816 | May 5, 1891. | T. H. Gilbert, | Buffalo, N. Y. |
| 459,873 | Sept. 22, 1891. | P. H. Hegerdahl, | Trondhjelm, Norway. |
| 465,031 | Dec. 15, 1891. | E. G. Scott, | Port Sunlight, Birkenhead, England. |
| 466,579 | Jan. 5, 1892. | C. F. and J. M. Simonin, | Philadelphia, Pa. |
| 471,323 | Mar. 22, 1892. | J. Castaneda, | Mexico, Mexico. |
| 475,106 | May 17, 1892. | G. L. P. Eyre and J. P. Hopkins, | London, England. |
| 481,502 | Aug. 23, 1892. | J. Massignan, | Paris, France. |
| 482,581 | Sept. 13, 1892. | C. Mann, | N. Y. City. |
| 482,661 | Sept. 13, 1892. | F. N. Turney, | Nottingham, England. |
| 482,995 | Sept. 20, 1892. | W. T. Cutter, | East Lynne, Conn. |
| 486,246 | Nov. 15, 1892. | J. W. Evans, | Cleveland, O. |
| 487,837 | Dec. 13, 1892. | H. Huvelle, | Waterford, Tex. |
| 493,158 | Mar. 7, 1893. | A. George, | Puteaux, France. |
| 494,647 | April 4, 1893. | C. W. Preston, | Brooklyn, N. Y. |
| 498,250 | May 17, 1892. | G. L. P. Eyre and J. P. Hopkins, | London, England. |
| 498,830 | June 6, 1893. | E. Watel, | Paris, France. |

| No. | Date. | Inventor. | Residence. |
|---|---|---|---|
| 509,589 | Dec. 28, 1893. | E. Watel, | Paris, France. |
| 515,240 | Feb. 20, 1894. | J. A. Lightall, | Syracuse, N. Y. |
| 515,889 | Mar. 6, 1894. | A. M. Todd, | Kalamazoo, Mich. |
| 516,769 | Mar. 20, 1894. | F. J. Machalski, | Brooklyn, N. Y. |
| 518,618 | April 24, 1894. | H. Mendes, | Paris, France. |
| 519,424 | May 8, 1894. | W. T. Forbes, | Atlanta, Ga. |
| 520,242 | May 22, 1894. | D. F. and W. E. Overton, | Solomons, Md. |
| 520,743 | May 29, 1894. | A. Schweitzer, | Chicago, Ill. |
| 522,130 | June 26, 1894. | F. N. Turney, | Nottingham, England. |
| 525,662 | Sept. 4, 1894. | J. Meikle, | Glasgow, Scotland. |
| 528,732 | Sept. 30, 1894. | W. O. Robbins, | N. Y. City. |
| 529,338 | Nov. 13, 1894. | E. E. Murphy, | Boston, Mass. |
| 530,893 | Dec. 11, 1894. | C. F. Hood, | Pittsburg, Pa. |
| 531,108 | Dec. 18, 1894. | W. T. Forbes, | Atlanta, Ga. |
| 532,799 | Jan. 26, 1895. | F. J. Machalski, | Brooklyn, N. Y. |
| 533,896 | Feb. 12, 1895. | E. Holthaus, | Canarsie, N. Y. |
| 533,940 | Feb. 12, 1895. | F. G. Wiselogel, | Indianopolis, Ind. |
| 534,602 | Feb. 19, 1895. | E. Fahrig, | Baltimore, Md. |
| 537,725 | April 16, 1895. | A. Schweitzer, | Chicago, Ill. |
| 540,256 | June 4, 1895. | B. Jaffe and L. Darmstaedter, | Martinickenfeld, Ger. |
| 545,271 | Aug. 27, 1895. | B. Dabney and W. J. Yopp, | Houston, Tex. |
| 545,900 | Sept. 10, 1895. | E. Maertens, | Providence, R. I. |
| 546,509 | Sept. 17, 1895. | E. Donard, | Rouen, France. |
| 546,708 | Sept. 24, 1895. | W. S. Pierce, | N. Y. City. |
| 548,958 | Oct. 29, 1895. | T. A. Clayton, | New Orleans, La. |
| 550,035 | Nov. 19, 1895. | H. A. Wheeler, | St. Louis, Mo. |
| 550,323 | Nov. 26, 1895. | E. Holthaus, | Canarsie, N. Y. |
| 550,506 | | | |
| 550,716 | Dec. 3, 1895. | H. Frasch, | Cleveland, O. |
| 550,628 | Dec. 3, 1895. | J. T. Lester, | Atlanta, Ga. |
| 551,396 | Dec. 17, 1895. | P. C. McIlhenny, | Jersey City, N. J. |
| 555,885 | Mar. 3, 1896. | O. Reichenbach, | London, England. |
| 556,065 | Mar. 10, 1896. | W. S. Pierce, | N. Y City. |

## *Rendering of Fats.*

These patents relate chiefly to the construction of apparatus consisting of vertical tanks with flat or conical bottoms, just above which is usually a perforated diaphragm. They are heated by steam jackets, or by coils of pipe in the tank. Many of them have attachments to carry off and deodorize the resulting offensive gases, as Nos. 46,103, 46,793, 52,640, 136,827, 138,954, 248,456, and reissues 2,048 and 2,385. 259,467 is a press; 137,698 and 241,119 show compressing rollers; 39,169 has a floating skimmer; 99,252 and 188,429 perforated cylinders; 288,106 relates to fish livers; while 242,171 and 255,863 include the use of aluminium chloride.

| No. | Date. | Inventor. | Residence. |
|---|---|---|---|
| 2,694 | June 27, 1842. | L. Montrop, | Baltimore, Md. |
| 3,784 | Oct. 9, 1844. | E. Wilson, | Cincinnati, O. |

| No. | Date | Inventor. | Residence |
|---|---|---|---|
| 3,827 | Nov. 13, 1844. | H. A. Amelung, | St. Louis, Mo. |
| 3,844 | Dec. 4, 1844. | A. Dunn, | Rotherhithe, England. |
| Reissue 167 | May 7, 1850. | E. Wilson, | Cincinnati, O. |
| 14,397 | Mar. 11, 1856. | F. Garcin, | Philadelphia, Pa. |
| 15,942 | Oct. 21, 1856. | J. J. Bates, | Brooklyn, N. Y. |
| 18,622 | Nov. 10, 1857. | A. Lapham, | " |
| 20,856 | July 13, 1858. | J. J. Bates, | " |
| 22,152 | Nov. 30, 1858. | J. L. Alberger, | Buffalo, N. Y. |
| 29,018 | July 3, 1860. | Tompert and Coyle, | Louisville, Ky. |
| 30,575 | Nov. 6, 1860. | J. M. Hunter, | New York, N. Y. |
| 32,633 | June 25, 1861. | H. S. Lewis, | Chicago, Ill. |
| 33,219 | Sept. 3, 1861. | J. Turner, | " |
| 39,169 | July 7, 1863. | Peck and Glover, | New York, N. Y. |
| 39,565 | Aug. 15, 1863. | C. E. Gray, | St. Louis, Mo. |
| 41,010 | Dec. 22, 1863. | J. J. Johnston, | Allegheny City, Pa. |
| 41,781 | Mar. 1, 1864. | S. Marsh, | Chicago, Ill. |
| 45,960 | Jan. 17, 1865. | T. Hopkins, | Cincinnati, O. |
| 46,103 | Jan. 31, 1865. | C. E. Gray, | New York, N. Y. |
| 46,204 | Feb. 7, 1865. | P. Andrew, | Cincinnati, O. |
| 47,264 | April 18, 1865. | W. Adamson, | Philadelphia, Pa. |
| 47,788 | May 23, 1865. | A. Black, | New York, N. Y. |
| 47,793 | Mar. 14, 1865. | C. E. Gray, | " |
| 48,559 | July 4, 1865. | D. H. Kaufman, | Kokams, Ind. |
| Reissue 2,048 | Aug. 8, 1865. | C. E. Gray, | New York, N. Y. |
| Reissue 2,049 | Aug. 8, 1865. | " | " |
| 52,640 | Feb. 13, 1866. | C. J. Everett, | " |
| 57,103 | Aug. 14, 1866. | Eckel & Schuyler, | " |
| 57,104 | Aug. 14, 1866. | " | " |
| 59,260 | Oct. 30, 1866. | W. H. Pinner, | " |
| 59,312 | Oct. 30, 1866. | C. J. Everett, | " |
| Reissue 2,385 | Oct. 30, 1866. | C. E. Gray, | " |
| 59,350 | Nov. 6, 1866. | W. Branigan, | Burlington, Ia. |
| Reissue 2,663 | June 25, 1867. | W. Perry, | North Bridgewater, Mass. |
| 62,439 | Feb. 26, 1867. | " | " |
| 69,897 | Oct. 15, 1867. | A. Broadnax, | New York, N. Y. |
| 80,006 | July 14, 1868. | C. C. and G. F. Pierson. | Philadelphia, Pa. |
| 81,743 | Sept. 1, 1868. | A. Broadnax, | New York, N. Y. |
| 81,744 | Sept. 1, 1868. | " | " |
| 82,195 | Sept. 15, 1868. | W. M. Bartram, | Philadelphia, Pa. |
| 87,482 | Mar. 2, 1869. | C. J. Everett, | New York, N. Y. |
| 96,315 | Nov. 2, 1869. | W. H. H. Glover, | Southold, N. Y. |
| 98,897 | Jan. 18, 1870. | L. Thorn, | New York, N. Y. |
| 99,250 | Jan. 25, 1870. | A. Smith, | Baltimore, Md. |
| 99,252 | Jan. 25, 1870. | " | " |
| 99,896 | Feb. 15, 1870. | W. J. Hooper and T. Hooper, | Baltimore, Md. |
| 100,063 | Feb. 22, 1870. | D. Pinger, | St. Joseph, Mo. |
| 103,234 | May 17, 1870. | J. W. Patterson, | New York, N. Y. |
| 124,983 | Mar. 26, 1872. | M. J. Stein, | " |
| Reissue 5,195 | Dec. 14, 1872. | A. Black, | " |
| 136,129 | Feb. 25, 1873. | A. Broadnax, | Mont Clair, N. J. |

| No. | Date. | Inventor. | Residence. |
|---|---|---|---|
| 136,208 | Feb. 25, 1873. | J. N. B. Bond, | New York, N. Y. |
| 136,420 | Mar. 4, 1873. | P. W. Dalton, | Jersey City, N. J. |
| 136,827 | Mar. 18, 1873. | H. S. Firman, | New York, N. Y. |
| 137,698 | April 18, 1873. | W. C. Marshall, | " |
| 138,954 | May 13, 1873. | S. Strunz, | Pittsburgh, Pa. |
| Reissue 5,643 | Nov. 4, 1873. | H. S. Firman, | New York, N. Y. |
| 146,586 | Jan. 20, 1874. | " | " |
| Reissue 5,925 | June 23, 1874. | A. Broadnax, | Mont Clair, N. J. |
| 153,874 | Aug. 4, 1874. | J. J. Willis, | New York, N. Y. |
| 168,611 | Oct. 11, 1875. | N. Caldwell, | Cincinnati, O. |
| 171,613 | Dec. 28, 1875. | H. Halvorson, | Cambridge, Mass. |
| 171,756 | Jan. 4, 1876 | P. Andrew, | Cincinnati, O. |
| 172,942 | Feb. 1, 1876. | " | " |
| Reissue 7,305 | Sept. 12, 1876. | J. L. Alberger, | Buffalo, N. Y. |
| 178,269 | June 6, 1876. | R. Bullymore. | " |
| 179,233 | June 27, 1876. | G. Upton, | Peabody, Mass. |
| 179,883 | July 18, 1876. | W. E. Andrew, | New York, N. Y. |
| 181,695 | Aug. 29, 1876. | A. S. Lyman, | " |
| 182,084 | Sept. 12, 1876. | A. L. Smith and A. Smith, Sr. | Cincinnati, O. |
| 188,429 | Mar. 13, 1877. | A. and L. Smith, | Cincinnati, O. |
| 189,541 | April 10, 1877. | H. Halvorson, | Cambridge, Mass. |
| 194,194 | Aug. 14, 1877. | A. Trotter, | Chicago, Ill. |
| 200,252 | Feb. 12, 1878. | R. Bullymore, | Buffalo, N. Y. |
| 204,471 | June 4, 1878. | T. J. Alberdingk, | Amsterdam, Holland. |
| 222,277 | Dec. 2, 1879. | A. Husbands, | St. Louis, Mo. |
| Reissue 9,254 | June 15, 1880. | H. Halvorson, | Cambridge, Mass. |
| 229,446 | June 29, 1880. | A. Miller and R. E. Gross, | New York, N. Y. |
| 231,558 | Aug. 24, 1880. | V. Gastard, | " |
| 233,417 | Oct. 19, 1880. | D. Jarves, | Detroit, Mich. |
| 241,119 | May 10, 1881. | O. F. Boomer, | Brooklyn, N. Y. |
| 242,171 | May 31, 1881. | V. R. von Ofenheim and R. von Haidinger, | Vienna, Austria. |
| 248,456 | Oct. 18, 1881. | T. and J. Hainsworth, | Chicago, Ill. |
| 251,088 | Dec. 20, 1881. | W. Bell, | New York, N. Y. |
| 251,105 | Dec. 20, 1881. | R. Neill, | Chicago, Ill. |
| 254,103 | Feb. 28, 1882. | G. Baylor, | St. Louis, Mo. |
| 254,279 | Feb. 28, 1882. | G. Cosine, | New York, N. Y. |
| 255,863 | April 4, 1882. | R. D. Fowler and W. J. Huet, | Paris, France. |
| 256,715 | April 18, 1882. | J. K. Martin, | Chicago, Ill. |
| 259,467 | Mar. 14, 1882. | T. Bray Bowers, | Chester, Pa. |
| 260,266 | June 27, 1882. | R. Whelan, | Chicago, Ill. |
| 261,259 | July 18, 1882. | W. Schneider, | Lehrberg, Bavaria, Ger. |
| 262,706 | Aug. 15, 1882. | F. Sulzberger, | New York, N. Y. |
| 264,189 | Sept. 12, 1882. | W. Mackler, | " |
| 267,634 | July 25, 1882. | F. Seltsam, | Forcheim, Bavaria, Ger. |
| 269,969 | Jan. 2, 1883. | W. Spiegel, | Newport, Ky. |
| 271,700 | Feb. 6, 1883. | R. Gerstang, | St. Louis, Mo, |
| 275,404 | April 10, 1883. | L. M. Ohly, | New York, N. Y. |
| 275,828 | April 17, 1883. | A. Kendrick and E. L. Jerauld, | Harwich, Mass. |

| No. | Date. | Inventor. | Residence. |
|---|---|---|---|
| 282,584 | Aug. 7, 1883. | S. Stephens, | Indianapolis, Ind. |
| 287,862 | Nov. 6, 1883. | C. H. Robinson, | Chicago, Ill. |
| 288,106 | Nov. 6, 1883. | E. Paysant, | Lockeport, Nova Scotia. |
| 291,535 | Jan. 8, 1884. | H. Rall, | St. Louis, Mo. |
| 295,184 | Mar. 18, 1884. | D. W. McElroy, | Keokuk, Iowa. |
| 299,821 | June 3, 1884. | " | " |
| 304,687 | Sept. 9, 1884. | M. Ams, | New York, N. Y. |
| 318,156 | May 19, 1885. | V. D. Anderson, | Cleveland, O. |
| 318,502 | May 26, 1885. | H. Rall, | St. Louis, Mo. |
| 318,503 | May 26, 1885. | " | " |
| 320,911 | June 30, 1885. | L. J. Caldwell, | Chicago, Ill. |
| 399,810 | Mar. 19, 1889. | F. Winter, | Allegheny, Pa. |
| 409,816 | Aug. 27, 1889. | V. D. Anderson, | Cleveland, O. |
| 422,260 | Feb. 25, 1890. | S. Stephens, | Indianapolis, Ind. |
| 425,314 | April 8, 1890. | M. A. Foster, | St. Louis, Mo. |
| 447,566 | Mar. 3, 1891. | J. Hobbs, | Boston, Mass. |
| 498,375 | May 30, 1893. | E. J. J. B. Benoit and J. Solary Vila, | Paris, France. |
| 477,023 | June 14, 1892. | A. Seiffert, | Frankenstein, Ger. |
| 507,222 | Oct. 24, 1893. | E. Holthaus, | Barren Island, N. Y. |
| 522,586 | July 10, 1884. | J. Cascaden, Jr., | Waterloo, Ill. |
| 524,012 | Aug. 7, 1894. | A. Wild, | Vienna, Austria. |
| 526,091 | Sept. 18, 1894. | A. White, | Minneapolis, Minn. |
| 528,718 | Nov. 16, 1894. | R. H. Doud, | Chicago, Ill. |
| 533,897 | Feb. 12, 1895. | E. Holthaus, | Canarsie, N. Y. |
| 533,898 | Feb. 12, 1895. | " | " |
| 539,055 | May 14, 1895. | " | " |
| 539,438 | May 25, 1895. | R. S. Merrill, | Wakefield, Mass. |
| 541,187 | June 18, 1895. | J. C. W. Stanley, | London, England. |
| 541,599 | June 25, 1895. | M. J. Dempsey, | Sydney, N. South Wales. |
| 543,350 | July 23, 1895. | H. Pfutzner, | Leipzig, Germany. |
| 543,384 | Sept. 23, 1895. | E. Holthaus, | Canarsie, N. Y. |
| 545,892 | Sept. 10, 1895. | " | " |
| 547,801 | Oct. 15, 1895. | H. F. C. Ott, | Altoona, Germany. |
| 548,793 | Oct. 29, 1895. | J. H. Winship, | N. Y. City. |
| 550,270 | Nov. 26, 1895. | B. F. Howland, | Philadelphia, Pa. |
| 550,342 | Nov. 26, 1895. | T. T. F. De La Choix, | Washington, D. C. |
| 555,551 | Mar. 3, 1896. | L. A. Addison, | Detroit, Mich. |
| 556,066 | Mar. 10, 1896. | W. S. Pierce, | N. Y. City. |

### *Decomposition of Fats.*

Decomposing fats relates chiefly to the manufacture of glycerine, and especially to the refining thereof, in which the following agents are used in the patents as indicated : Aluminium, 345,174 ; calcium salts, 280,894 : barium salts, 280,893 ; zinc, 267,753, 278,849, 293,344, 336,795 ; silicates, 371,127 ; tin, 272,510 ; alcohol, 2,534 ; oxalic acid, 276,551 ; oxide of copper, 277,575 ; oxides of alkalies, 359,148 ; oxide of lead, 263,915 ; sodium chloride, 264,616 ; electricity, 284,862 ; by atomizer, 258,614 ; by steam, 309,515 and 316,104 ; by centrifuge, 251,992 ; forming sulpho-sebacic acid, 243,377 ; and making fatty acids by sulphuric acid, 290,835. The patents of Hageman and Mitting are important in relation to glycerine.

| No. | Date. | Inventor. | Residence, |
|---|---|---|---|
| 2,534 | April 1, 1842. | J. H. Smith, | Brooklyn, N. Y. |
| 11,766 | Oct. 3, 1854. | R. A. Tilghman, | Philadelphia, Pa. |
| 21,711 | Oct. 5, 1858. | M. Work, | Cincinnati, O. |
| 22,691 | Jan. 25, 1859. | J. C. Appenzeller, | " |
| 22,765 | Jan. 25, 1859. | Wright and Fouché, | Paris, France. |
| 28,315 | May 15, 1860. | R. A. Tilghman, | Philadelphia, Pa. |
| 76,974 | April 21, 1868. | R. C. Barton, | Brooklyn, N. Y. |
| 144,000 | Oct. 28, 1873. | C. F. A. Simonin, | Philadelphia, Pa. |
| 167,607 | Sept. 14, 1875. | T. M. Fell, | Brooklyn, N. Y. |
| 201,704 | Mar. 26, 1878. | F. Sahlfeld, | New York, N. Y. |
| 215,372 | May 13, 1879. | A. Kuehne, | Jersey City, N. J. |
| 242,272 | May 31, 1881. | C. V. Clolus, | Paris, France. |
| 242,702 | June 7, 1881. | F. Seltsam, | Forcheim, Germany. |
| 243,377 | June 28, 1881. | A. M. Jacobs, | Moscow, Russia. |
| 251,992 | Jan. 3, 1881. | J. P. Battershall, | New York, N. Y. |
| 255,504 | Mar. 28, 1882. | H. Heckel, | Cincinnati, O. |
| 255,505 | Mar. 28, 1882. | " | " |
| 256,613 | April 18, 1882. | B. T. Babbitt, | New York, N. Y. |
| 256,614 | April 18, 1882. | " | " |
| 262,913 | Aug. 22, 1882. | F. Armandy, | Saint Fons, France. |
| 263,915 | Sept. 5, 1882. | J. K. Kessler, | Milwaukee, Wis. |
| 264,616 | Sept. 19, 1882. | E. L. M. Brochon, | Milan, Italy. |
| 266,504 | Oct. 24, 1882. | T. J. O. Farrel, | Dublin, Ireland. |
| 267,753 | Nov. 21, 1882. | V. Litzschmann, | New York, N. Y. |
| 272,510 | Feb. 20, 1883. | M. Ams and V. Litzelman, | " |
| 275,976 | April 17, 1883. | B. T. Babbitt, | " |
| 276,551 | May 1, 1883. | J. B. Battershall, | " |
| 277,575 | May 15, 1883. | J. P. Kessler, | Milwaukee, Wis. |
| 278,849 | June 5, 1883. | E. O. Beaujard, | Aubervilliers, France. |
| 280,893 | July 10, 1883. | " | " |
| 280,894 | July 10, 1883. | " | " |
| 282,547 | Aug. 7, 1883. | W. F. C. McCarty, | Berlin, Germany. |
| 283,266 | Aug. 14, 1883. | " | " |
| 283,936 | Aug. 28, 1883. | T. G. Walker, | Morristown, N. J. |
| 284,862 | Sept. 11, 1883. | M. H. Lackersteen, | Chicago, Ill. |
| 286,448 | Oct. 9, 1883. | O. Laist, | Cincinnati, O. |
| 290,835 | Dec. 25, 1883. | J. A. T. Bang and J. De Castro, | Paris, France. |
| 293,344 | Feb. 2, 1884. | E. F. Michaud and E. N. Michaud, | Auberville, France. |
| 306,832 | Oct. 21, 1884. | F. H. Houghton, | New York, N. Y. |
| 309,515 | Dec. 23, 1884. | E. O. Beaujard, | " |
| 316,104 | April 21, 1885. | B. T. Babbitt, | " |
| 319,971 | June 16, 1885. | R. Gleberman, | Chicago, Ill. |
| 336,795 | Feb. 23, 1886. | J. H. Clinton, | St. Louis, Mo. |
| 345,174 | July 6, 1886. | C. L. Porter, | " |
| 359,148 | Mar. 8, 1887. | R. Glebermann, | Chicago, Ill. |
| 371,127 | Oct. 4, 1887. | A. Domeier and Otto Christian Hageman, | London, England. |
| 384,725 | June 19, 1888. | P. Stickney, | Madeira. |
| 385,367 | July 3, 1888. | E. K. Mitting, | Chicago, Ill. |
| 385,105 | June 26, 1888. | A. Domier and O. C. Hageman, | London, England. |

| No. | Date. | Inventor. | Residence. |
|---|---|---|---|
| 392,819 | Nov. 13, 1888. | M. Highley, | Jersey City, N. J. |
| 399,028 | Mar. 5, 1889. | A. W. Trench, | Brooklyn, N. Y. |
| 406,158 | July 2, 1889. | C. Bardy, | Paris, France. |
| 413,616 | Oct. 22, 1889. | A. Domier and O. C. Hageman, | London, England. |
| 413,617 | " " | " " | " " |
| 413,618 | " " | " " | " " |
| 413,619 | " " | " " | " " |
| 414,709 | Nov. 12, 1889. | G. and E. Janssen, | Chicago, Ill. |
| 419,610 | Jan. 14, 1890. | E. K. Mitting, | " |
| 420,830 | Feb. 4, 1890. | C. L. Porter, | " |
| 428,468 | May 20, 1890. | A. Domier and O. C. Hageman, | London, England. |
| 428,469 | " " | " " | " " |
| 428,470 | " " | " " | " " |
| 428,471 | " " | " " | " " |
| 428,484 | " " | O. C. Hageman, | London, England. |
| 428,509 | " " | E. K. Mitting, | Chicago, Ill. |
| 428,510 | " " | " | " |
| 428,566 | " " | " | " |
| 431,986 | July 8, 1890. | " | " |
| R.11,125 | Nov. 18, 1890. | " | " |
| 443,761 | Dec. 30, 1890. | O. C. Hageman, | London, England. |
| 452,507 | May 19, 1891. | J. Patten, | N. Y. City. |
| 453,829 | June 9, 1891. | A. Domeier and O. C. Hageman, | London, England. |
| 453,834 | " " | O. C. Hageman, | " " |
| 453,835 | " " | " | " " |
| 458,647 | Sept. 1, 1891. | J. Van Ruymbeke, | Chicago, Ill. |
| 458,648 | " " | " " | " " |
| 465,474 | Dec. 22, 1891. | O. C. Hageman,<br>E. K. Mitting, | London, England.<br>Chicago, Ill. |
| 475,757 | May 31, 1892. | Domeier & Hageman, | London, England. |
| 475,758 | " " | " " | " " |
| 481,879 | Aug. 30, 1892. | R. O. Unglaub, | Pendleton, England. |
| 510,734 | Dec. 12, 1893. | A. F. Trageser, | N. Y. City. |
| 522,132 | June 26, 1894. | J. Van Ruymbeke, | Chicago, Ill. |
| 522,133 | " " | " " | " |
| 522,156 | June 26, 1894. | F. L. Dyer, | Washington, D. C. |
| 522,998 | July 17, 1894. | E. K. Mitting, | Chicago, Ill. |
| 544,366 | Aug. 13, 1895. | H. J. Morrison, | Clifton, O. |

### *Refining of Fat Oils.*

This class relates chiefly to the use of chemical agents in brightening oils, as follows: Mercuric chloride, 2,855; sodium silicate and calcium chloride, 365,921; fuller's earth, 233,453 and 345,872; sulphuric acid and hydrocarbon, 347,345; copper sulphate, 294,940; sodium chloride, 295,633; potassium chromate and manganese dioxide, 306,324 and 316,663; nitric acid, 292,699; sodic hydrate, 301,783; carbon dioxide, 331,702, 328,024, 342,692; electricity, 336,622; potassium bichromate alone, 169,008; air-blast, 273,036, 312.351 and 366,991; spraying, 278,116, 353,358; thin films on plates, 153,867, 354,755;

bone black, 237,484, 278,187. The following patents relate to the subjects specified, viz.: Cocoa-nut oil, 230,518, 356,398; wool-oil, 271,192; porpoise oil, 340,851; palm oil, 281,823; coloring oil, 284,811, 304,390; jellifying petroleum, 241,505; menthol, 337,716; use of sodium peroxide, 543,079.

| No. | Date. | Inventor. | Residence. |
|---|---|---|---|
| No number | Nov. 17, 1808. | E. Fourestier, | Philadelphia, Pa. |
| | July 20, 1829. | C. E. Ruggles, | New York, N. Y. |
| 2,600 | April 30, 1842. | E. Marsk, | Alton, Ill. |
| 2,855 | Nov. 21, 1842. | A. Trabaud, | “ |
| 4,439 | April 4, 1846. | P. Fitzsimmons, | West Troy, N. Y. |
| 7,767 | Mar. 12, 1850. | T. Antisell, | “ |
| 14,012 | Jan. 1, 1856. | Philo Marsh, | South Adams, Mass. |
| 47,333 | April 18, 1865. | T. L. Robinson, | Boston, Mass. |
| 153,867 | Aug. 4, 1874. | F. Walton, | Staines, Great Britain. |
| 169,008 | Oct. 19, 1875. | J. P. Kinney, | Brooklyn, N. Y. |
| 184,341 | Nov. 14, 1876. | H. A. Clark, | Boston, Mass. |
| 195,297 | Sept. 18, 1877. | J. Mayer, | New York, N. Y. |
| 222,983 | Dec. 30, 1879. | D. D. Cattenach, | Providence, R. I. |
| 230,518 | July 27, 1880. | A. P. Ashbourne, | Boston, Mass. |
| 233,453 | Oct. 19, 1880. | A. W. Winter, | San Francisco, Cal. |
| 237,484 | Feb. 8, 1881. | R. A. Chesebrough, | New York, N. Y. |
| 240,910 | May 3, 1881. | A. Koellner, | Neu Muehlen, Kiel, Prus. |
| 241,505 | May 17, 1881. | C. J. T. DuMotay, A. J. Rossi, A. Bourgignon, Paul Casamajor. | New York, N. Y. |
| 242,171 | May 31, 1881. | V. R. von Oppenheim and R. R. von Heidinger, | Vienna, Austria. |
| 247,141 | Sept. 13, 1881. | J. J. Williams, | St. Louis, Mo. |
| 249,868 | Nov. 22, 1881. | P. Andrew, | Cincinnati, O. |
| 250,830 | Dec. 13, 1881. | W. R. Meeds, | Newark, O. |
| 251,629 | Dec. 27, 1881. | Wm. Hara, | Chicago, Ill. |
| 252,516 | Jan. 17, 1882. | F. M. Reed, | Lawrenceburg, Ind. |
| 253,821 | Feb. 14, 1882. | J. McDougall, | London, England. |
| 256,220 | April 11, 1882. | R. Irvine, | Roylston, Granton, Scot. |
| 266,859 | Aug. 31, 1882. | W. R. Meeds, | Newark, O. |
| 271,192 | Jan. 25, 1883. | O. Braun and O. Liebreich, | Berlin, Germany. |
| 273,036 | Feb. 27, 1883. | J. Davis, | Brooklyn, N. Y. |
| 277,331 | May 8, 1883. | P. G. Oster, | Köln, Prussia. |
| 278,166 | May 22, 1883. | W. J. Morrison, | Nashville, Tenn. |
| 278,187 | May 22, 1883. | H. Selink, | Ludwigshafen on Rhein, Germany. |
| 281,823 | July 24, 1883. | B. T. Babbitt, | New York, N. Y. |
| 282,970 | Aug. 16, 1883. | S. M. Eiseman, | “ |
| 284,811 | Sept. 11, 1883. | H. R. Burk, | Brooklyn, N. Y. |
| 287,216 | Aug. 30, 1883. | J. A. Bang and C. A. Sanguinetto, | Marseilles, France. |
| 287,627 | Aug. 30, 1883. | R. C. Clark and W. T. Beecher, | Corry, Pa.<br>Cleveland, O. |
| 290,784 | Nov. 25, 1883. | H. McManus, | Brooklyn, N. Y. |

| No. | Date. | Inventor. | Residence. |
|---|---|---|---|
| 292,669 | Jan. 29, 1884. | W. F. C. M. McCarty, | Aix-la-Chapelle, Ger. |
| 294,940 | Mar. 11, 1884. | P. C. Vogellus, | Gloucester, Mass. |
| 294,978 | Mar. 11, 1884. | S. M. Eiseman, | New York, N. Y. |
| 295,633 | Mar. 25, 1884. | J. H. Gray, | Brooklyn, N. Y. |
| 299,904 | June 3, 1884. | J. A. W. Borchers, | West Medford, Mass. |
| 301,783 | Feb. 8, 1884. | E. S. Wilson, | Camberwell, Surrey. England. |
| 304,390 | Sept. 2, 1884. | R. A. Williams and J. Bragg, | Cleveland, O. |
| 306,324 | Oct. 7, 1884. | J. Davis, | Boston, Mass. |
| 312,351 | Feb. 17, 1885. | J. W. and F. R. Hoard, | Providence, R. I. |
| 313,693 | Mar. 10, 1885. | J. C. Thornton, | Mt. Vernon, O. |
| 316,633 | April 28, 1885. | A. Rock, | Chihuahua, Mexico. |
| 318,410 | May 19, 1885. | J. B. Armstrong, | Chicago, Ill. |
| 327,871 | Oct. 6, 1885. | C. M. Ely, | Lexington, Ky. |
| 328,024 | Oct. 13, 1885. | J. B. Grant, | Brooklyn, N. Y. |
| 330,613 | Nov. 17, 1885. | D. J. Ogilvy, | Cincinnati, O. |
| 331,702 | Dec. 1, 1885. | J. B. Grant and A. Mason, | Brooklyn, N. Y. |
| 335,957 | Feb. 9, 1886. | G. J. Pilkington, | Chester, Pa. |
| 336,622 | Feb. 23, 1886. | C. L. Coffin, | Detroit, Mich. |
| 337,459 | Mar. 9, 1886. | J. C. Thornton, | Mt. Vernon, Ind. |
| 337,716 | Mar. 9, 1886. | A. M. Todd, | Nottawa, Mich. |
| 340,851 | April 27, 1886. | A. P. Ashburner, | Philadelphia, Pa. |
| 342,692 | May 25, 1886. | J. B. Grant, | Brooklyn, N. Y. |
| 344,275 | June 22, 1886. | F. H. Jacobs and J. Y. Lockwood, | St. Louis, Mo. |
| 345,872 | July 20, 1886. | W. B. Albright, | Boston, Mass. |
| 347,345 | Aug. 17, 1886. | Allan T. Hall, | Hull, England. |
| 353,358 | Nov. 30, 1886. | J. W. and F. R. Hoard, | Providence, R. I. |
| 354,755 | Dec. 21, 1886. | F. Walton, | Twickenham, Middlesex, England. |
| 356,398 | Jan. 18, 1887. | A. P. Ashburner, | Philadelphia, Pa. |
| 365,921 | July 5, 1887. | R. Hunt and E. S. Wilson, | Lancaster, England. |
| 366,991 | July 19, 1887. | A. T. Andrews, | Avon, Conn. |
| 377,406 | Feb. 7, 1888. | G. W. Scollay, | N. Y. City. |
| 378,113 | Feb. 21, 1888. | | |
| 378,114 | | | |
| 396,636 | Jan. 22, 1889. | G. B. Wiggin, | South New Market, N. H. |
| 396,774 | Jan. 29, 1889. | A. Sommer, | Berkeley, Cal. |
| 405,852 | June 25, 1889. | G. W. Scollay, | N. Y. City. |
| 414,722 | Nov. 12, 1889. | C. H. Robinson, | Philadelphia, Pa. |
| 414,723 | “ “ | “ | “ |
| 414,724 | “ “ | “ | “ |
| 432,251 | July 15, 1890. | A. Smith, | Brockley, England. |
| 439,515 | Oct. 28, 1890. | R. Hunt, | Liverpool, England. |
| 446,117 | Feb. 10, 1891. | G. Race, | Norwich, N. Y. |
| 451,531 | May 5, 1891. | A. Sommer, | Berkeley, Cal. |
| 452,967 | May 26, 1891. | T. H. Gray, | Bermondsey, England. |
| 455,286 | June 30, 1891. | H. Kellogg, | New Haven, Conn. |
| 461,675 | Oct. 20, 1891. | W. A. Grant, | Houston, Texas. |
| 463,875 | Nov. 24, 1891. | A. Sommer, | Berkeley, Cal. |
| 467,992 | Feb. 2, 1892. | W. N. Hartley, | Dublin, Ireland. |
| | | W. E. B. Blenkinsop, | London, England. |

| No. | Date. | Inventor. | Residence. |
|---|---|---|---|
| 468,498 | Feb. 9, 1892. | G. H. Weiss,<br>E. H. Dickerson, | Jersey City, N. J.<br>N. Y. City. |
| 468,937 | Feb. 16, 1892. | A. Sommer, | Berkeley, Cal. |
| 470,714 | Mar. 5, 1892. | " | " |
| 470,715 | " " | " | " |
| 474,864 | May 17, 1892. | G. W. Scollay, | N. Y. City. |
| 496,987 | May 9, 1893. | W. N. Blakeman, | Mt. Vernon, N. Y. |
| 496,988 | " " | " | " |
| 496,989 | " " | " | " |
| 496,990 | " " | " | " |
| 496,991 | " " | " | " |
| 498,162 | May 23, 1893. | W. D. Field, | Milburn, N. Y. |
| 498,821 | June 6, 1893. | G. W. Scollay, | N. Y. City. |
| 498,822 | June 6, 1893. | " | " |
| 529,727 | Nov. 27, 1894. | William Griscom, | Bound Brook, N. J. |
| 532,682 | Jan. 15, 1895. | W. Mills, | London, England. |
| 536,163 | Mar. 19, 1895. | F. N. Turney, | Nottingham, England. |
| 538,828 | May 7, 1895. | A. Gentzsch, | Vienna, Austria. |
| 542,758 | April 16, 1895. | G. A. Freiss, | Mülhausen, Germany. |
| 543,079 | July 23, 1895. | F. Burton, | London, England. |
| 547,122 | Oct. 1, 1895. | R. G. Latting, Jr., | Brownwood, Texas. |
| 556,914 | Mar. 10, 1896. | F. Marohn, | Milwaukee, Wis. |
| 557,098 | Mar. 31, 1896. | F. B. Aspinwall, | London, England. |
| 557,457 | Mar. 31, 1896. | F. Walton, | " " |

### *Bleaching of Fats and Oils.*

Color is removed by sunlight from wax, 190,995 and 194,799; and by steam and water, 167,607; by sodium carbonate, potassium bichromate, and sulphuric acid, 167,607 and 216,558; by charcoal and boneblack, 171,328, and from spruce gum by filtration, 57,304.

| No. | Date. | Inventor. | Residence. |
|---|---|---|---|
| 57,304 | Aug. 21, 1866. | H. B. Estey, | Houlton, Maine. |
| 71,768 | Dec. 3, 1867. | T. Leonhard, | Paterson, N. J. |
| 136,881 | Mar. 16, 1873. | D. D. Templeton, | New York, N. Y. |
| 171,328 | Dec. 21, 1875. | J. Tardos, | New Orleans, La. |
| 189,867 | April 24, 1877. | R. Macdonald, | " |
| 190,995 | May 22, 1877. | H. T. Yaryan, | Richmond, Ind. |
| 194,799 | Sept. 4, 1877. | " | " |
| 216,558 | June 17, 1878. | Jos. Davis, | Philadelphia, Pa. |

### *Lard Coolers.*

Lard coolers consist of apparatus whereby the lard is made to have a fine even grain, which is obtained by agitation or stirring while cooling. This is effected by stirrers of various shapes moving around in a tub, like an ice-cream freezer, as in patents Nos. 9, 21, 32. Or, secondly, by frame stirrers moving back and forth in a long trough, as Nos. 11, 22, 35. Thirdly, by spiral stirrers in long tanks, of which character are the following numbers: 8, 10, 15, 18, 28, 31, 33, 43. Fourthly, by coiled pipes carrying the fat through tanks of water, or by the pipes carrying the water through the tanks of lard, as Nos. 1, 4, 24, 34, 38. And finally, by blowing cold air directly from the open mouths of pipes through the material, as in Nos. 20, 23, 29. This class also includes such machines as are used in cooling artificial butter, as No. 37.

| | No. | Date. | Inventor. | Residence. |
|---|---|---|---|---|
| 1 | 37,120 | Dec. 9, 1862, | W. J. Wilcox, | New York, N. Y. |
| 2 | 37,469 | Jan. 20, 1863. | " | " |
| 3 | 40,644 | Nov. 17, 1863. | V. E. Rusco, | Chicago, Ill. |
| 4 | 43,352 | June 28, 1864. | G. B. Turrell, | New York, N. Y. |
| 5 | 46,476 | Feb. 21, 1865. | A. G. Knapp, | " |
| 6 | 47,361 | April 18, 1865. | A. R. Judson, | " |
| 7 | 51,075 | Nov. 21, 1865. | G. C. Napheys, | Philadelphia, Pa. |
| 8 | 61,907 | Feb. 5, 1867. | G. B. Williams, | New York, N. Y. |
| 9 | 78,925 | June 16, 1868. | G. C. Cassard, | Baltimore, Md. |
| 10 | Reissue 3,183 | Nov. 10, 1868. | G. B. Williams, | New York, N. Y. |
| 11 | Reissue 3,196 | Nov. 17, 1868. | W. J. Wilcox, | " |
| 12 | 85,117 | Dec. 22, 1868. | J. N. Merriam, | Cambridgeport, Mass. |
| 13 | Reissue 3,358 | April 6, 1869. | G. C. Cassard, | Baltimore, Md. |
| 14 | 107,621 | Sept. 20, 1870. | M. Nolden, | Prussia. |
| 15 | 109,548 | Nov. 22, 1870. | E. F. Ring, | St. Louis, Mo. |
| 16 | 119,818 | Oct. 10, 1871. | A. E. Camp and C. L. Reid, | Louisville, Ky. |
| 17 | 125,162 | April 2, 1872. | O. J. Backus, | San Francisco, Cal. |
| 18 | 130,534 | Aug. 13, 1872. | E. F. Ring, | St. Louis, Mo. |
| 19 | 131,847 | Oct. 1, 1872. | G. C. Cassard, | Baltimore, Md. |
| 20 | 142,744 | Nov. 9, 1873. | A. Smith, | Cincinnati, O. |
| 21 | 146,852 | Jan. 27, 1874. | W. J. Wilcox, | New York, N. Y. |
| 22 | 151,941 | June 9, 1874. | A. M. Warthman, Jr., | Philadelphia, Pa. |
| 23 | 152,208 | June 23, 1874. | R. B. Bancroft, | Boston, Mass. |
| 24 | 161,703 | April 6, 1875. | F. C. Pray, | New York, N. Y. |
| 25 | 165,617 | July 13, 1875. | F. Rockstroh, | Baltimore, Md. |
| 26 | 167,636 | Sept. 14, 1875. | G. Bogen, Jr., | Cincinnati, O. |
| 27 | 168,610 | Oct. 11, 1875. | N. Caldwell, | " |
| 28 | 169,180 | Oct. 26, 1875. | J. G. C. Lee, | New York, N. Y. |
| 29 | 170,935 | Dec. 14, 1875. | R. Bullymore, | Buffalo, N. Y. |
| 30 | 179,809 | July 11, 1876. | C. Meister, | Baltimore, Md. |
| 31 | 189,032 | April 3, 1877. | W. P. Harvey, | " |
| 32 | 195,535 | Sept. 25, 1877. | H. Rieman, | " |
| 33 | 219,423 | Sept. 9, 1879. | C. J. Yergason, | |
| 34 | 220,811 | Oct. 21, 1879. | P. Cudahy, | Milwaukee, Wis. |
| 35 | 228,691 | June 8, 1880. | G. A. Stanley, | Cleveland, O. |
| 36 | 251,512 | Dec. 27, 1881. | W. Bell, | New York, N. Y. |
| 37 | 259,783 | June 20, 1882. | W. Tunstill, | " |
| 38 | 277,797 | May 15, 1883. | J. Siddely and F. N. Mackey, | Liverpool, England. |
| 39 | 280,025 | June 26, 1883. | F. Fiederlein, | New York, N. Y. |
| 40 | 284,212 | Sept. 4, 1883. | E. Kells, | Cleveland, O. |
| 41 | 287,362 | Oct. 23, 1883. | O. G. Burnham, | Chicago, Ill. |
| 42 | 289,809 | Dec. 11, 1883. | " | " |
| 43 | 291,692 | Jan. 8, 1884. | S. H. Cochran, | Everett, Mass. |
| 44 | 426,914 | April 29, 1890. | D. C. Camp, | Gainesville, Ga. |
| 45 | 469,305 | Feb. 23, 1892. | B. Rosenthal and O. D. Noble, | Chicago, Ill. |

| | No. | Date. | Inventor. | Residence. |
|---|---|---|---|---|
| 46 | 490,505 | Jan. 24, 1893. | H. Sartorius, | St. Louis, Mo. |
| 47 | 494,841 | April 4, 1893. | T. Burkhardt, | Brooklyn, N. Y. |
| 48 | 512,642 | Jan. 9, 1894. | W. L. Whitman, | Bridgeport, Pa. |
| 49 | 518,618 | April 24, 1894. | H. Mendes, | Paris, France. |

## *Artificial Butter.*

The Mège patents are the principal ones on oleomargarine: 338,538 relates to rancid butter; 344,730 to testing for butter fats; 266,777–8 to cotton seed oil and flour; 262,207 to slippery elm; 226,467 to phosphoric acid. and 519,580 to the manufacture of an edible fat from petroleum, and 266,417 to butter color.

| No. | Date. | Inventor. | Residence. |
|---|---|---|---|
| 68,639 | Sept. 10, 1867. | D. H. McGregory, | Detroit, Mich. |
| 80,506 | July 28, 1868. | L. S. Robbins, | |
| 110,626 | Jan. 3, 1871. | H. W. Bradley, | Binghamton, N. Y. |
| 120,026 | Oct. 17, 1871. | " | Plainfield, N. J. |
| 121,162 | Nov. 21, 1872. | L. L. A. E. Picot de la Peyrouse, | Paris, France. |
| 142,891 | Sept. 16, 1873. | G. Bogart, W. H. Cramer, W. L. Lewis, | Laporte City, Iowa. |
| 145,840 | Dec. 23, 1873. | J. R. Brown, | New Haven, Conn. |
| 146,012 | Dec. 30, 1873. | H. Mège, | Paris, France. |
| 148,767 | Mar. 17, 1874. | B. Smith, | San Francisco, Cal. |
| R.5,868 | May 12, 1874. | H. Mège, | Paris. France. |
| 152,999 | Aug. 11, 1874. | W. E. Andrew, | New York City. |
| 154,251 | Aug. 18, 1874. | J. Hobbs, | Boston, Mass. |
| 154,372 | Aug. 25, 1874. | Churchill & Englehart, | New York City. |
| 163,610 | May 25, 1875. | J. C. Rorick, | Wauseon. O. |
| 166,955 | Aug. 24, 1875. | W. E. Andrew, | New York City. |
| 169,008 | Oct. 19, 1875. | J. P. Kinney, | Brooklyn, N. Y. |
| R.6,737 | Nov. 16, 1875. | W. E. Andrew, | New York City. |
| 173,581 | Feb. 15, 1876. | Garret Cosine, | " |
| 187,327 | Feb. 13, 1877. | A. Springer, | Cincinnati, O. |
| 198,334 | Dec. 12, 1877. | R. W. Barnard, | Fayette, Ia. |
| R.8,048 | Jan. 22, 1878. | W. E. Andrew, | New York City. |
| R.8,424 | Sept. 24, 1878. | H. Mège, | Paris, France. |
| R.10,137 | June 13, 1882. | Com. Mfrg. Co., | New York City. |
| 264,714 | Sept. 19, 1892. | H. Lauferty, | " |
| 265,833 | Oct. 10, 1882. | " | " |
| 226,467 | April 13, 1880. | F. F. Wilkins, | London, England. |
| 236,483 | Jan. 11, 1881. | O. Boyesen, | Buffalo, N. Y. |
| R.9,892 | Oct. 11, 1881. | T. F. Wilkins, | London, England. |
| 253,820 | Feb. 14, 1882. | G. W. Kendall and R. R. Robert, | New Orleans, La. |
| 258,992 | June 6, 1882. | S. H. Cochran, | Everett, Mass. |
| 262,207 | Aug. 8, 1882. | " | " |
| R.10,181 | Aug. 1, 1882. | " | " |
| 263,042 | Aug. 22, 1882. | J. Hobbs, | Boston, Mass. |
| 263,199 | Aug. 22, 1882. | N. I. Nathan, | New York City. |
| 264,545 | Sept. 19, 1882. | G. S. Marshall, | Everett, Mass. |
| 264,546 | Sept. 19, 1882. | W. Cooley, | Waterbury, Vermont. |

| No. | Date. | Inventor. | Residence. |
|---|---|---|---|
| 266,417 | Oct. 24, 1882. | H. Berthold, | New York City. |
| 266,508 | Oct. 24, 1882. | G. H. Webster, | Chicago, Ill. |
| 266,580 | Oct. 24, 1882. | W. H. Burnett, | " |
| 266,777 | Oct. 31, 1882. | O. H. Coumbe, R. R. Roberts, | New Orleans, La. |
| 266,788 | Oct. 31, 1882. | O. H. Coumbe, R. R. Roberts, | New Orleans, La. |
| 267,037 | Nov. 14, 1882. | H. R. Wright, | Albany, N. Y. |
| 270,454 | Jan. 9, 1883. | J. H. McDonald, | New York City. |
| 271,239 | Jan. 30, 1883. | J. Hobbs, | Boston, Mass. |
| 271,240 | " " | " | " |
| 271,241 | " " | " | " |
| 271,242 | " " | " | " |
| 271,243 | " " | " | " |
| 271,244 | " " | " | " |
| 278,100 | May 22, 1883. | J. Morrison, | Nashville, Tenn. |
| 285,878 | Oct. 2, 1883. | S. H. Cochran, | Everett, Mass. |
| 285,973 | Oct. 2, 1883. | " | " |
| 285,974 | Oct. 2, 1883. | " | " |
| 286,778 | Oct. 16, 1883. | A. J. Chase, | Boston, Mass. |
| 289,100 | Nov. 27, 1883. | J. Hobbs, | " |
| 295,180 | Mar. 18, 1884. | G. Lawrence, | London, England. |
| 299,085 | June 3, 1884. | S. Schwarzschild, | New York City. |
| 327,630 | Oct. 6, 1885. | E. J. Woodruff, | Chicago, Ill. |
| 336,324 | Feb. 16, 1885. | C. A. Johnson, | Stockholm, Sweden. |
| 336,438 | Feb. 16, 1886. | G. W. Sample, | York, Pa. |
| 338,538 | Mar. 23, 1886. | C. Marchand, | New York City. |
| 344,730 | June 20, 1886. | L. Fagerstein, | Chicago, Ill. |
| 346,002 | July 20, 1886. | E. J. Oatman, | Dundee, Ill. |
| 367,940 | Aug. 9, 1887. | G. W. Towar, | Detroit, Mich. |
| 378,381 | Feb. 31, 1888. | " | " |
| 430,536 | June 17, 1890. | D. McGregory, | Los Angeles, Cal. |
| 489,814 | Jan. 10, 1893. | D. W. Hudson, C. D. Bunch, T. La Strong, | " |
| 515,523 | Feb. 27, 1894. | P. F. Gibbons, T. Buchanan, | Hebron, Ill. |
| 519,980 | May 15, 1894. | A. W. Winter, | Chicago, Ill. |
| 520,513 | May 29, 1894. | W. N. Blakeman, Jr., | New York City. |
| 522,409 | July 3, 1894. | J. H. Filbert, | Baltimore, Md. |
| 526,322 | Sept. 18, 1894. | J. Lister, | Chicago, Ill. |
| 530,298 | Dec. 4, 1894. | D. H. Burrill, | Little Falls, N. Y. |
| 535,792 | Mar. 12, 1895. | J. H. Filbert, | Baltimore, Md. |
| 550,676 | Dec. 3, 1895. | T. Cordeweener and A. D. Kunwald, | Paris, France. |
| 551,134 | Dec. 10, 1895. | C. Morfit, | London, England. |

## *Suint.*

Suint is the peculiar fat obtained by washing wool. It is a kind of animal soap, and contains a large amount of potassium salts, which are often separated. The pharmaceutical products known as lanolin, etc., consist of purified wool fat.

| No. | Date. | Inventor. | Residence. |
|---|---|---|---|
| 271,192 | Jan. 23, 1883. | O. Braun and O. Liebreich, | Berlin, Germany. |
| 299,904 | June 3, 1884. | J. A. W. Borchers, | W. Bedford, Mass. |
| 389,295 | Sept. 11, 1888. | W. Graf Lesum, | Bremen, Germany. |
| 399,906 | Mar 19, 1889. | H. W. Langbeck, | Loughton, Essex, Eng. |
| 443,940 | Dec. 30, 1890. | M. L. Griffin, E. S. Thayer, | Holyoke, Mass. |
| | | E. S. Buffum, | Salem, Mass. |
| 451,642 | May 5, 1891. | I. Roos, | Frankfort-on-Main, Germany. |
| 475,395 | May 24, 1892. | R. B. Griffin, | Auburndale, Mass. |
| 511,051 | Dec. 19, 1893. | J. C. Lahusen, | Bremen, Germany. |
| 539,386 | May 14, 1895. | W.D. Hartshorne and Emile Maertens, | Providence, R. I. |
| 539,388 | May 14, 1895. | Emile Maertens, | " |
| 540,256 | June 4, 1895. | B. Jaffe and L. Darmstaedter, | Martinickenfeld, Ger. |
| 545,899 | Sept. 10, 1895. | Emile Maertens, | Providence, R. I. |
| 548,942 | Oct. 20, 1895. | J. H. Wingfield, | Mont Clair. N. J. |
| 549,041 | Oct. 20, 1895. | " | " |
| 549,042 | " " | " | " |
| 553,222 | Jan. 21, 1896. | J. C. Anderson, | Highland Park, Ill. |

## *Wax.*

The first six patents relate to sealing wax, and the last two to grafting wax. No. 177,347 to paraffin, 303,436 and 441,366 to resin hardened by lime, the so-called "Adamanta," and 308,958 and 321,090 to bees-wax.

| No. | Date. | Inventor. | Residence. |
|---|---|---|---|
| 3,544 | April 17, 1844. | J. Tatman, | Philadelphia, Pa. |
| 131,684 | Sept. 24, 1872. | E. L. and F. Hyde, | Baltimore, Md. |
| 132,329 | Oct. 15, 1872. | A. Smith and W. Stuart, | Cincinnati, O. |
| 143,771 | Oct. 21, 1873. | W. J. Lumb, | Zanesville, O. |
| 143,886 | Oct. 21, 1873. | J. L. Baker. | |
| 147,818 | Feb. 24, 1874. | W. H. Butler, | Brooklyn, N. Y. |
| 165,942 | July 27, 1875. | L. R. Mears, | New York City. |
| 172,391 | Jan. 18, 1876. | W. R. Clough, | Newark, N. J. |
| 177,347 | May 16, 1876. | J. B. Meriam, | Cleveland, O. |
| 179,759 | July 11, 1876. | Ray and Wm. C. Blakiston, | Quebec, Canada. |
| 190,681 | May 15, 1877. | C. F. Herman, | St. Louis, Mo. |
| 194,799 | Sept. 4, 1877. | H. T. Yaryan, | Richmond, Ind. |
| 266,858 | Oct. 31, 1882. | T. McLoughlin, | Judsonville, Cal. |
| 274,484 | Mar. 27, 1883. | P. C. Ghaler, | York, Pa. |
| 288,525 | Nov. 13, 1883. | J. Waterous, | Brooklyn, N. Y. |
| 296,921 | April 15, 1884. | C. H. Bundy, | Middletown, O. |
| 303,436 | Aug. 12, 1884. | A. Kissel, | Frankfort-on-Main Germany. |
| 305,981 | Sept. 30, 1884. | J. F. Thomson, | Rockland, Mass. |
| 306,486 | Oct. 14, 1884. | G. W. Hastings, | Grafton, Mass. |
| 307,746 | Nov. 11, 1884. | G. W. Coddington, | Middletown, O. |
| 308,958 | Dec. 9, 1884. | J. D. Enas, | Napa City, Cal. |
| 321,090 | June 30, 1885. | " | " |

| No. | Date | Inventor. | Residence |
|---|---|---|---|
| R.10,714 | April 20, 1886. | A. Kissel, | Frankfort-on-Main, Germany. |
| 389,898 | Sept. 25, 1888. | R. M. Perrine, | Cleveland, O. |
| 421,904 | Feb. 25, 1890. | W. Bruening, | E. Orange, N. J. |
| 440,762 | Nov. 18, 1890. | E. Roche, | Providence, R. I. |
| 441,366 | Nov. 25, 1890. | A. Kissel, | Salem, Mass. |
| 483,512 | Sept. 27, 1892. | J. C. Eckert, | Dayton, O. |
| 527,218 | Oct. 9, 1894. | A. S. Rutherford, | New York City. |
| 527,219 | " " | " | " |
| 545,940 | Sept. 10, 1895. | S. M. Brooks, | Burlington, Conn. |
| 545,941 | " " | " | " |

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